

**PRACTICAL
INORGANIC CHEMISTRY**

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PRACTICAL INORGANIC CHEMISTRY

Adapted to meet the Requirements of the
I Sc. Standard of the Universities of
Calcutta and Allahabad

By

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CALCUTTA

CHUCKERVERTY, CHATTERJEE & Co.

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Santanu Kumar Banerji

PREFACE

This short Practical Course is meant as a companion volume to my "Elementary Inorganic Chemistry." It is based mainly upon the Syllabus drawn up for the guidance of the I.Sc. Practical Class.

A few subjects which could only be summarily treated in the systematic course have necessarily received here more ample attention; *e.g.*, experimental demonstrations of Boyle's and Gay-Lussac's Laws, and of structure of flame as also acidimetry and alkalimetry, etc.

In the preparation of this little book I have been deeply indebted to the unremitting and assiduous labours of Messrs. Gopibhusan Sen, B.A., and Pabitra Kumar Dutt, M.A., B.Sc., Demonstrator and Assistant respectively in the Chemical Department. Mr. Chandrabhusana Bhaduri, B.A., Senior Demonstrator, has also favoured me with many valuable suggestions. I avail myself also of this opportunity to express my hearty thanks to Mr. Prabodha Chandra Chattopadhyay, M.A., of the Analytical Department of the Bengal Chemical and Pharmaceutical Works, Limited, for his careful revision of the work while it was going through the press and the painstaking care with which he has looked over the proofs.

PRESIDENCY COLLEGE, }
July 1st., 1911.

P. C. RAY.

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COMPLETE LIST OF APPARATUS REQUIRED FOR EACH STUDENT.

(TO BE HAD OF THE BENGAL. CHEMICAL AND
PHARMACEUTICAL WORKS, LD., CALCUTTA.)

N. B. Those marked with an asterisk() may be
used by a batch of students.*

Bunsen Burner	...	1
Rose top	1
Batswing burner	1
* Blowpipe for table	1
* Foot blower	1
Triangular file, 4" long	1
Cork borer, set of three	..	1 set
* Cork press, crocodile shape	...	1
* Chemical balance (with agate knife • edges)	1
* Sartorius Balance	1
* Measuring cylinder, 250 c.c.	...	1
Porcelain basin, 8 c/m.	...	2
Glass tube, assorted	2 lb.
Glass rod 6"	2
Flask, flat bottom, 500 c.c.	...	2
Cork, assorted	$\frac{1}{4}$ gross.
* Metre rule	1
Measuring flask, 1000 c.c.	...	1

List of Apparatus

* Weight box, ordinary, 50 gr.	...	1
* Do Sartorius, 50 gr.	...	1
* Burette, stop., 50 c.c.	...	1
* Do unstoppered, with jet and clip 25 c.c.	...	1
* Stand and clamp for above	...	1
* Thermometer, 110°C. and 212°F.	...	1
* Do 360°C.	...	1
Beakers, assorted	...	3
Funnels, assorted	...	3
Funnel stand	...	1
Filter paper, diam. 9 c/m.	...	100
Wire gauze, 4" sq.	...	1
Rubber tubing, bore 3 m/m.	...	1 yd.
Do do 5 m/m.	...	2 yd.
Do do 7 m/m.	...	1 yd.
Tripod stand	...	1
Test tube	...	12
Crystallising dish	...	1
Retort, stopd., 125 c.c.	...	1
Receiver for do.	...	1
Retort stand with 3 rings	...	1
Pneumatic trough	...	1
Distilling flask 60 c.c.	...	1
* Liebig's condenser, 40 c/m.	...	1
Water bath	...	1
Weighing bottle, stopd.	...	1
Squared paper	...	2 sheets
Crucible with lid	...	1

List of Apparatus

xv

Pipe clay triangle	1
Hard glass test tube	1
* Retort clamp, small and large	1 each
Deflagrating spoon	1
Gas jars	3
Cover for do.	3
Taper holder	1
* Barometer tube	1
* Mercury trough, porcelain	1
* Copper flask	1
Bent pipette	1
Boyle's Law apparatus	1
Coefficient of expansion of air apparatus	1
* Hofmann clip	1
* Mohr's clip	1
Round bottom flask, 1½ litre	1
Rubber cork for do.	1
* Kipp's apparatus, 1 litre	1
Mortar and pestle, 8 c/m	1
Magnet, 4" long	1
Woulffe's bottle, 2 necks	3
Thistle funnel 30 c/m.	1
Do 35 c/m.	1
* Apparatus to show that hydrogen is lighter than air	1
Calcium chloride tube, straight, 15 c/m. long	1
Voltameter, simple	1

List of Apparatus

Calcium chloride tube, bent, with moisture trap	1
Porcelain boat	.	.	1
* Air bath	..		1
* Desiccator	...		1
Watch glass, 45 m/m	1
Pear shaped round bottom flask	.		1
Round bottom flask 500 c.c.	...		1
* Sulphuric acid apparatus	.		1
* Gas tube, grad., 100 c.c.	...		1
* Tall jar for immersing above	...		1
* Teclu Burner	1
* Combustion tube	1
U tubes with side tube	...		1
Sample tubes	...		3
Detonating bottle			1
Lamp chimney	.		1
Mouth blowpipe			1
Blowpipe jet for Bunsen Burner	..		1
Asbestos board, 4" sq.			1
* Separating funnel, 60 c.c.			1
Platinum wire	1 inch.
<hr/>			
Approximate value	R. 500

PRACTICAL INORGANIC CHEMISTRY



CHAPTER I BURNERS AND THEIR USES

The beginner should first acquire some knowledge of the burners commonly used in a laboratory as sources of heat and learn how to use them.

THE BUNSEN BURNER

Take a Bunsen burner (Fig. 1) and examine its different parts which may be readily unscrewed. Make a neat sketch in your note book. Notice carefully the air holes near the base of the burner and how they may be regulated by turning a movable brass piece. In some burners the tube itself is movable. Fit up the burner again by screwing its parts and light it with the air holes closed; an ordinary luminous flame will be produced. Hold a porcelain basin over it and notice that soot is at once deposited. Now open the air holes and observe the smokeless non-luminous flame. The gas

Rose Burner—Batswing Burner

gas escapes from the small orifice into the wider vertical tube, draws air with it through the air holes and the

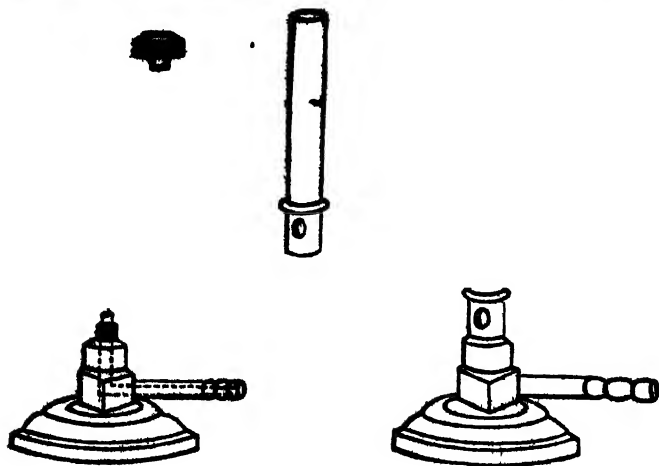


Fig. 1.

mixture of gas and air burns at the top with a non-luminous flame, which is intensely hot. This burner is used where high temperature is required.

ROSE BURNER

Take a rose top, fix it on the top of a Bunsen burner and light it. Observe that the gas burns in a number of minute jets. Rose tops are commonly used for heating large surfaces where it is not advisable to heat a particular point strongly.

THE BATSWING BURNER

This is an ordinary burner resembling that commonly used for illuminating purposes. It is without air holes.

Light it and observe that a flat luminous flame like a fish tail is produced (Fig. 2). Hence it is also called a fish-tail

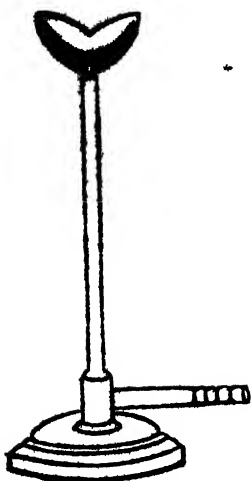


Fig. 2.

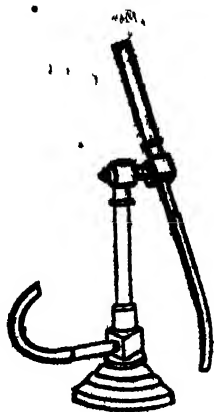


Fig. 3.

burner. This burner is very convenient for heating long lengths of glass tubes and therefore always used for bending glass tubes. It does not produce a very high temperature like a Bunsen burner. Moreover, the soot produced in a batwing flame is rather advantageous as it protects glass from sudden cooling and thereby unpleasant cracks are avoided.

BLOWPIPE FOR GLASS BLOWERS

Examine a table blowpipe (Fig. 3) and observe that it consists of two concentric metallic tubes, the outer one is connected with the gas supply and the other with a foot

blower. Light the gas; it burns with a luminous flame. Now send in a blast of air from the foot blower; the gas burns with a non-luminous flame. Notice that when the gas supply is reduced and the foot blower worked cautiously, a *pointed flame* (Fig. 4) is produced which is most suitable for joining glass tubes. On the other hand, when a large supply of gas is turned on and a considerable blast of air forced into the flame a *brush flame* (Fig. 5) is produced which is most suitable for blowing bulbs and sealing the ends of hard glass tubes.

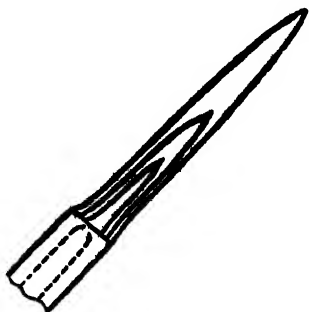


Fig. 4.

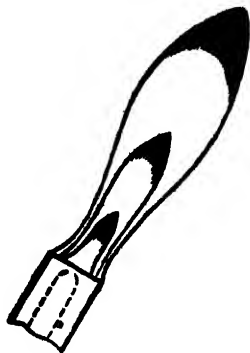


Fig. 5.

HOW TO CUT A PORTION OFF A GLASS TUBING

Take a triangular file and make a sharp scratch with it across the tube at the point where it is to be cut off. Now hold the tube with both hands, with the scratch between them and opposite the thumbs (see Fig. 6) and break it asunder by trying to pull the ends apart, and at the same time applying a gentle pressure with

the thumbs as if to bend it away from the portion scratched. In this way the parts will be separated without any rough projecting edges.

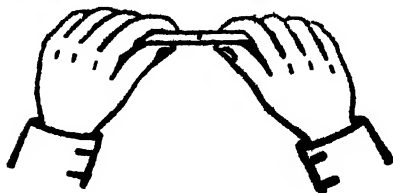
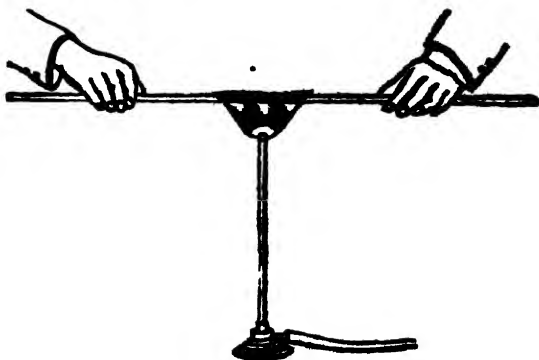


Fig. 6.

The sharp edges should always be rounded off by carefully heating the end in a Bunsen flame for a few seconds till the glass softens.



• Fig. 7.

HOW TO BEND A GLASS TUBE

Take a glass tube about 10 inches long, hold it horizontally with both hands and heat a length of about .

Stirring Rod—Glass Jet

2 inches of it in a Bunsen flame as shown in Fig. 7. Rotate it constantly so as to heat it evenly all round. When it has properly softened take it out of the flame and bend it to a right angle. The bend should be even and in the same plane with the two arms (Fig. 8. A).

Try to bend another piece of tube in a Bunsen flame. As only a short length of it is heated, the bend is never uniform (Fig. 8. B).

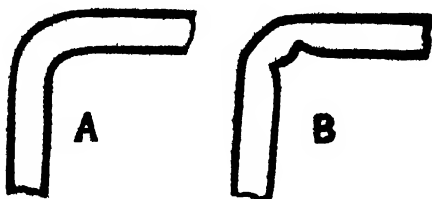


Fig. 8.

HOW TO MAKE A GLASS STIRRING ROD

Take a glass rod, and cut off a portion about 8 inches long by making a sharp scratch with a triangular file, as directed for cutting glass tubes. Fuse its two ends in a non-luminous flame, turning round and round, till the sharp edges are softened and smooth.

HOW TO MAKE GLASS JETS

Take a piece of glass tube and heat it in a Bunsen flame, or better in a blow-pipe flame, twirling it continually. When quite soft, remove the tube from the source of heat and pull the two ends apart, at first gently and then more rapidly until the tube is contracted to a very

narrow bore. Hold the tube vertically till it cools down to be rigid enough to retain its shape. When cold, cut off into two jets. Round off the point of the jet, taking

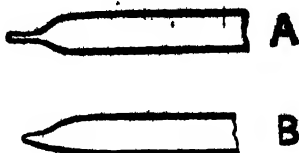


Fig. 9.

care that it may not be sealed. In Fig. 9, A shows the form of a jet properly done, while B shows a defective one.

HOW TO SEAL THE END OF A GLASS TUBE

Heat a glass tube in a blowpipe flame turning it round until it softens; draw it out before it hardens. Now take one of these drawn out tubes and take off as much of the tail as possible after having melted it once again. Next hold the narrow end of the tube in the flame, turning it round all the while until a small quantity of the glass has collected and then blow into it as gently as will give only a rounded end of the same thickness and size as the body of the tube.

HOW TO BLOW A BULB AT THE END OF A TUBE

Seal the end of a tube as before and heat the closed end in a small brush flame of the blow-pipe—turning it round and round so as to avoid the bulging of the tube in any way. At last when the glass has been

properly softened and thickened as will make a bulb of the size required, remove it quickly from the flame and blow into it steadily rotating it all the time. If the form of the bulb is not regular melt the glass and blow again. Finally the bulb should not be allowed to cool all at once. It should be properly annealed by being held in a luminous flame and moved about for a minute or two.

HOW TO FIT UP A WASH BOTTLE

After having acquired some practice in such operations as glass cutting, bending and blowing, the student may now proceed with the fitting up of a wash bottle

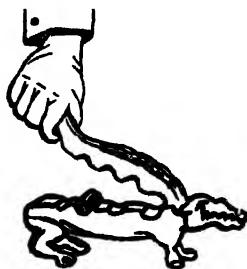


Fig. 10.

Take a flask with flat bottom, of about half a litre capacity. Select a sound cork which is just too large to fit into its neck. In order to render it air tight soften it by pressing in a cork press (Fig. 10) or by wrapping a piece of paper round it and gently rolling under foot. The cork, when pressed, should fit air tight into the neck of the flask. Now bore two holes in the cork by

means of a cork borer (Fig. 11). In doing so select a borer, the diameter of which is almost equal to or



Fig. 11.

slightly smaller than that of the tubing you want to fit in the cork. Place the sharp edge of the borer on the cork, hold it parallel to the axis and rotate slowly while exerting a gentle pressure all the time. When the borer is almost through, work it more carefully so as not to tear the cork at the other end. Be careful that the two holes be parallel to and slightly apart from each other and not inclined to the axis of the cork.

Having bored the cork, take a long glass tube and from it cut off two pieces of the required lengths and see that they fit air tight in the cork, if the holes have been slightly too small for the tubes, enlarge them by means of a rat-tail file. One of the two tubes should be approximately double that of the other and when fitted into the neck of the flask should almost reach its bottom.

Now bend the two tubes in a batswing flame, the longer tube to an angle of about 50° and the shorter one to an angle of about 130° , so that when they are fitted together in the wash bottle the two arms outside should be in one straight line. Round off the edges of the

tubes and fit them in the cork as shown in Fig. 12. Next make a fine jet about 2 inches long and attach it



Fig. 12.

to the longer tube by means of a piece of rubber tubing as shown in the sketch. The jet can thus be moved freely in all directions. Cleanse the flask thoroughly and fill it with distilled water and fit the cork into its neck. The wash bottle is now ready for use.

CHAPTER II

SYSTEM OF MEASUREMENTS

The unit of length universally used for scientific measurements is a *metre*, equivalent to 39·37 inches. This was first selected at the end of the eighteenth century as being of a convenient length for practical measurements and because it was supposed to bear a natural relation to the size of the earth, being a definite fraction of the length of the earth's circumference through the poles.

Subsequently, however, the idea of this supposed relation was given up and now the metre is legally defined as the exact distance between two marks on a platinum rod preserved in Paris.

To be in harmony with the decimal system of numeration, the unit of length has been sub-divided into tenths, hundredths, and thousandths on one side and multiplied by ten, hundred and thousand on the other. Thus we have,

1 Millimetre	(mm)	=	0·001 metre
1 Centimetre	(cm)	=	0·01 „
1 Decimetre	(dc m.)	=	0·1 „
1 Metre	(M.)	=	1 „
1 Dekametre	(Dkm)	=	10 „
1 Hectometre	(Hm)	=	100 „
1 Kilometre	(Km)	=	1000 „

Unit of Surface

The unit of surface is obtained by squaring any of the units of length given above. The most commonly

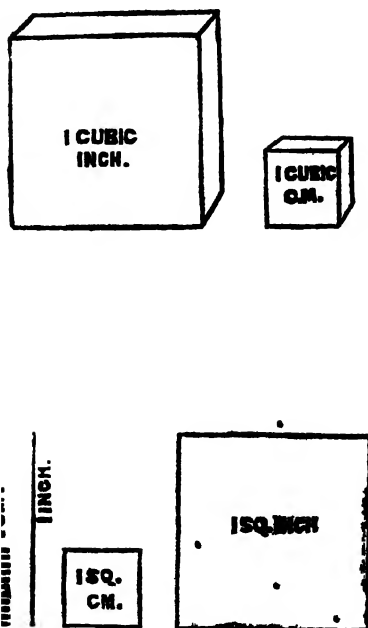


Fig. 13

used is the square centimetre (sq. cm.), being a surface equal in area to a square of 1 cm. side (Fig. 13).

For measuring volumes, the *litre* is taken to be the unit, and is equal to the volume of a cube, each face of which has an area of 1 sq. decimetre. If such a cube were made of very thin metal sheet it would contain 1 litre of water. One cubic centimetre (1 c.c.) is one thousandth part of a litre by volume.

The unit of *mass* or *weight* is the gram. This was primarily defined as the weight of one cubic centimetre of pure water at the temperature when it is heaviest (*i. e.* at 4°C.).

Now, however, the gram is defined as the thousandth part of a mass of platinum kept in Paris, constructed in accordance with the above relation, *i.e.*, equal in weight to 1000 c.c. or 1 litre of water at its maximum density point.

The gram has also been divided according to the decimal system. Thus we have,

1 Milligram	(mgm.)	= 0.001 gram.
1 Centigram	(cgm.)	= 0.01 „
1 Decigram	(dcgm.)	= 0.1 „
1 Gram or Gramme	(gm.)	= 1 „

The multiples are—

1 Decagram	(D)	= 10 grams.
1 Hectogram	(H)	= 100 „
1 Kilogram	(K or Kg)	= 1000 „

1 pound = 453.593 grams.

1 gram = 15.432 grains.

MEASUREMENT OF MASS

THE BALANCE

The balance is an instrument used to compare the masses of two bodies.

An ordinary chemical balance, such as is shown in Fig. 14, consists of two scale pans, P and P', sus-

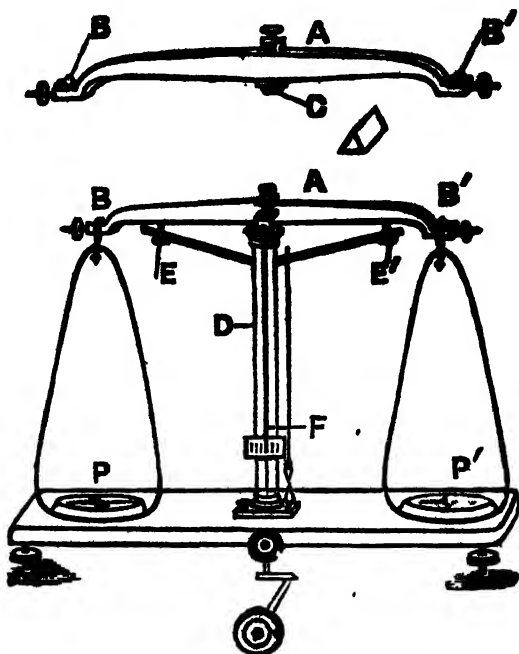


Fig. 14

pended from two agate knife edges B and B' at the ends of a brass beam A. The beam also carries another

knife edge C, at the centre, by means of which it rests upon an agate plane on the top of the pillar D. This agate plane can be raised or lowered by turning the milled head in front of the balance. When not in use the agate plane is lowered and the beam rests on two pegs E and E'. At the two ends of the beam are two small movable screws by which the balance can be adjusted. The pointer F is attached to the centre of the beam and at right angles to it and moves before a scale fixed to the pillar. When the balance is rightly adjusted, the pointer should move over equal divisions on each side of the zero of the scale. If not, the swinging should be set right by moving the screws at the ends of the beam. To protect the instrument from dust and draughts it is enclosed in a glass case.*

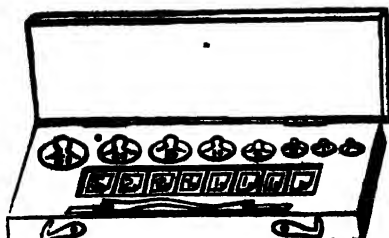


Fig. 15

THE WEIGHT BOX

In an ordinary weight box (Fig. 15) the larger weights from 100 grams down to 1 gram are made of brass some-

* The teacher should explain the different parts of the balance and show the students how it is adjusted.

times nickel-plated. The fractional weights ranging from 0.5 gram down to 0.01 gram are made of german silver or of aluminium. Sometimes the numbers stamped on the smaller weights give their values in milligrams; thus,

that marked 500 = 0.5 gram.
 „ „ 200 = 0.2 „
 „ „ 50 = 0.05 „
 and so on.

The figure shows the general arrangement of the weights in the box

Figure 16 represents a very accurate form of chemical balance (Sartorius). The weights up to 1 centigram or 10 milligrams are placed in the pan and below that a bent piece of platinum wire, called the *rider*, is used which when placed on different parts on the top of the beam give different values. Each half of the beam is graduated into 50 parts starting from zero in the middle. The rider when placed at one end of the beam weighs 1 centigram and when placed on division 25 it weighs, $25 \times 0.0002 = 0.005$ gram. Thus each division shows a difference of 0.0002 gram

DIRECTIONS FOR WEIGHING

1. Before beginning to weigh, see that the balance pans are clean. If not, have them dusted off by means of a camel hair brush. Then raise the beam and see that the pointer swings over equal divisions on both sides of the zero of the scale. ° If not, adjust it as directed.

2. Never put anything on or take anything out from the pans when the balance is in swing.

3. Never put the thing to be weighed directly on the pans, but use a watch glass or a weighing bottle to hold it.

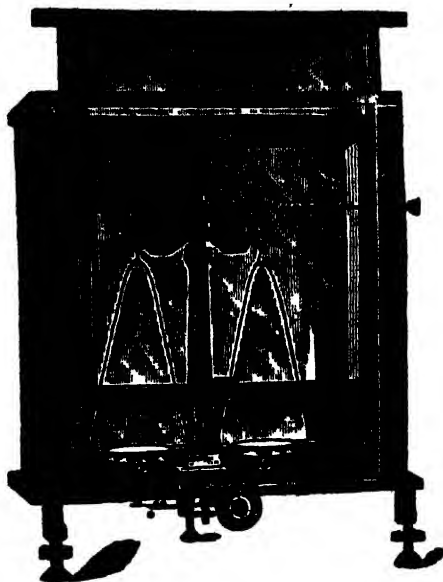


Fig. 16.

4. Never handle the weights with your fingers, but always with the pair of forceps provided for this purpose.

5. Never weigh anything when it is hot.

6. Put the thing to be weighed on the left pan P, and on the right one P' place a weight which seems to be heavier than the object. Raise the beam and note the direction in which the pointer moves. If the right

pan is heavier, replace the weight by the next lower one. If this be too great, repeat the process with successive weights until the weight in P' is less than the weight in P . Then go on adding the lower weights in the above manner until the balance is in equilibrium, which is indicated by the swinging over equal divisions of the scale on both sides.

Now write down the values of the weights in your note book and add up. Remove the weights from the pan and keep them back in their proper places in the box and check your result while doing so.

MEASUREMENT OF VOLUMES

Liquids are generally measured by volume and the value expressed in terms of a litre or in cubic centimetre. For this purpose certain glass vessels are used which are graduated empirically, the numbers etched on them indicating their capacity.

Four different forms of these, *e.g.*, measuring cylinder, measuring flask, pipette and burette are in common use in the laboratory.



Fig. 17.

MEASURING CYLINDER

The form is shown in Fig. 17. It is graduated throughout its entire length in c.c. or sometimes in

fractions of it. The number against the topmost mark indicates its total capacity.

MEASURING FLASK

These flasks are made to contain a certain volume of a liquid when filled up to a mark on the neck. Various sizes of these are in use, e.g., litre, half litre, quarter litre and 100 c.c. The one in Fig. 18 is marked 1000 c.c., *i.e.*, it will contain 1 litre of water when filled up to the mark on the neck.

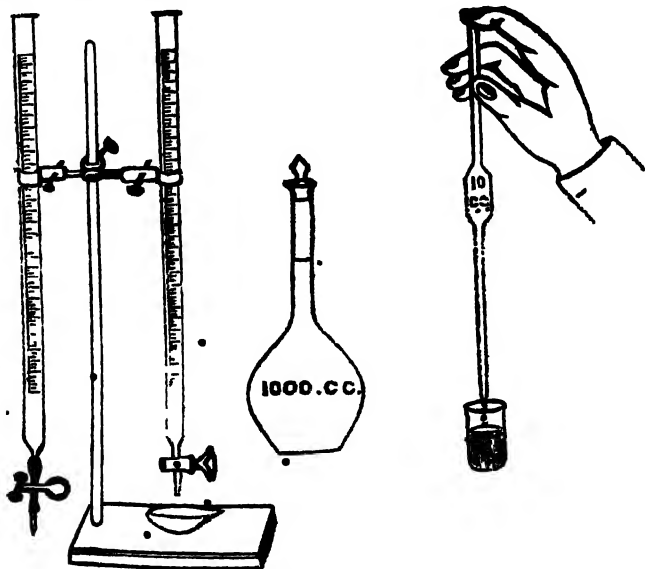


Fig. 18.

PIPETTE

It is a glass tube, drawn to a point at one end, with a cylindrical bulb at its middle. It is manipulated

by holding it between the thumb and the other three fingers except the index finger which is free. The liquid is sucked in with the mouth until its level is above the mark on the stem, but at a safe distance from the mouth, when the index finger is pressed on the top. Now release the pressure of your finger and allow the liquid to run out drop by drop until the lower meniscus of the liquid is just at the mark. The pipette now contains the amount of liquid marked on it which can be delivered to a beaker by removing the index finger. Allow the liquid to drain to the last drop by holding the tip against the side of the beaker, but do not blow out the liquid.

BURETTE

It is a glass tube drawn out at one end and closed by a stopcock or connected to a jet by means of a piece of rubber tubing and closed by a clip. It is graduated in c.c. and in fractions of a c.c., almost throughout its entire length. Those in general use have a capacity of 50 c.c. graduated into one-tenths. For use, clamp it upright as shown in Fig. 18. Fill it with the liquid by means of a small funnel (which is removed after filling it) until the level of the liquid is considerably above the topmost or zero mark. Now open the stopcock or the clip, so that the liquid may sweep out the air contained in the jet, and when the lower meniscus of the liquid is at the zero mark, close the tap. The burette is now ready and any accurately

measured volume of liquid can be delivered by opening the tap.

READING THE VOLUME IN GRADUATED VESSELS

Owing to capillary attraction the liquid in a vessel assumes a curved surface which is called the meniscus. In reading a burette, for instance, hold a sheet of white paper behind it and the meniscus will appear as a dark

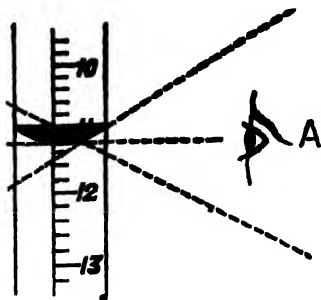


Fig. 19.

line. Now take the reading from the lower edge of this dark line, taking care that your eye, the lower edge of the meniscus and the mark on the burette are all in one straight line. The correct position of the eye is shown in the figure at A (Fig. 19).

MEASUREMENT OF TEMPERATURE

The temperature of a body is generally measured by a simple instrument called the *thermometer*. The most



Fig. 20.

ordinary form of thermometer used in laboratories consists of a capillary glass tube having a small bulb blown at one end. The bulb and a portion of the stem are filled with mercury. The mercury is then boiled to drive off the air and the open end closed hermetically by fusing the glass (Fig. 20).

It is graduated according to different systems. In the Centigrade system, the lower fixed point is the temperature of melting ice which is denoted by zero. The higher fixed point is the temperature at which pure water boils under ordinary conditions. The space between these two points is divided into 100 divisions, each division is called a *degree*. The Centigrade scale is generally adopted in laboratories.

In the Fahrenheit scale, the zero point is 32 degrees below the melting point of ice and the space between the melting point of ice and the boiling point of water is divided into 180 divisions. Thus the temperature of boiling water is $(180 + 32) 212^{\circ}\text{F}$. Hence, 1 Fahrenheit degree $= \frac{5}{9}$ degree Centigrade. The Fahrenheit scale is generally used for clinical and meteorological purposes.

The temperature of one scale can be converted into that of the other according to the following formulae :—

Centigrade to Fahrenheit :

$$\frac{C^{\circ} \times 9}{5} + 32 = F^{\circ}.$$

Fahrenheit to Centigrade :

$$\frac{(F^{\circ} - 32) \times 5}{9} = C^{\circ}.$$

CHAPTER III

SIMPLE CHEMICAL OPERATIONS WITH WATER

Take some muddy water in a beaker or some clear water in which a quantity of soft clay has been added to make it turbid. Set aside a portion of the muddy water for twenty four hours and proceed with the other portion as follows :—

Take an ordinary circular filter paper, fold it into four segments and open out one. Fit it into the form



Fig. 21.

of a cone in a funnel, moisten it with a few drops of pure water and press the cone air-tight into the funnel.

Support the funnel in a filter stand. Now hold a glass rod against the edge of the containing beaker and pour the muddy water down the rod on the thicker fold of the filter paper (Fig. 21). Collect the liquid as it passes through the filter in another beaker placed below the funnel. The stem of the funnel should touch the side of the collecting beaker in order to avoid the spattering of the liquid. If the liquid is not clear in one operation this process should be repeated till it is perfectly clear.

The above process of separating the suspended solid particles from a liquid is called *filtration*.

Take the clear liquid in a porcelain basin, place it on a piece of wire gauze supported over the ring of a stand or a tripod and heat it by a Bunsen flame. Evaporate



Fig. 22.

the liquid to a small bulk and finally finish the operation over boiling water in a water bath, the simplest form of which consists of a beaker half filled with water and

placed on a wire gauze over a tripod (fig. 22). A thin layer of solid residue is left at the bottom of the basin.

Thus you learn that fine particles of solid which are in a state of suspension are alone removed by filtration; whereas substances in solution cannot thus be separated.

Examine the water which has been allowed to settle for twenty four hours. Notice that the water is perfectly clear and a thin layer of mud is deposited at the bottom of the vessel. Decant a portion of the clear water, taking care not to disturb the sediment. Evaporate it in a basin as before; a solid residue is left behind.

SEPARATION OF THE INGREDIENTS OF A MIXTURE OF CHALK AND NITRE

Take some chalk powder and mix it with a quantity of nitre. Weigh out about 5 grams of the mixture in a clean beaker. Add some water and heat for some time. Set aside the liquid for a few minutes in order that the suspended particles may settle down. In the mean time weigh an ordinary filter paper and fit it into a funnel. Now decant the clear liquid to the filter paper and collect the filtrate in another beaker. When most of the clear liquid has been poured off, add some more hot water and after stirring allow it to settle. Again decant the clear liquid to the same filter and transfer the whole of the residue from the beaker to the filter

Separation of Chalk and Nitre

by means of a jet of water from the wash bottle. Care should be taken that not a trace of the substance is left. Finally wash the residue in the filter several times by means of a jet of hot water.

Now remove the funnel with the filter paper and leave it in a steam oven to dry.

Transfer the filtrate into a weighed basin and rinse the beaker into the basin two or three times with a jet of water. Place the basin on a water bath and evaporate the liquid to dryness. Now allow it to cool and wipe the bottom of it with a clean piece of cloth and weigh. Put the basin again on the water bath, cool and weigh as before. Repeat the process until the consecutive weights are constant. The difference in weight gives the amount of nitre.

Take out the funnel from the steam oven and allow it to cool and weigh the paper with the chalk powder.

Calculate your results as follows.

Weight of mixture	=	4.85	grams.
Weight of basin	=	24.34	"
Do. + nitre	=	26.52	"
∴ Weight of nitre	=	2.18	"
Weight of filter paper	=	1.32	"
Do. + chalk	=	3.97	"
∴ Weight of chalk	=	2.65	"
Weight of nitre	=	2.18	"
		<hr/>	
		4.83	"
Difference	=	0.02	"

TO PROVE THE PRESENCE OF DISSOLVED
GAS IN WATER

Take a beaker and place in it an inverted funnel which would almost cover the bottom of the beaker. Fill it with water so that the stem of the funnel should be under water. Also fill a test tube with water, cover its mouth with your thumb and invert it on the stem of the funnel as shown in figure 23, taking care to expel all air bubbles from the tube. Apply heat by a Bunsen burner and observe the bubbles of gas rising from

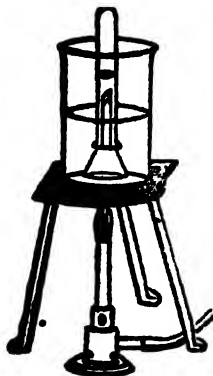


Fig. 23.

the heated water and collecting in the test tube. When no more bubbles are observed, cool the water and examine the gas. The gas might be tested for carbon dioxide or air by the usual tests.

CRYSTALLISATION

Take some nitre and dissolve it in a certain volume of water in a basin. Add more nitre, a little at a time,

stirring well with a rod. Ultimately, you will find that some nitre will remain undissolved, because the water cannot dissolve any more nitre at the temperature of the solution. The solution is now said to be saturated at that temperature. Warm the solution and observe that it will dissolve more and more of the substance. It is thus evident that the point of saturation also rises with the temperature.

Take this clear solution in a glass dish, and allow it to cool slowly. Nitre separates out in the solid state, assuming certain definite geometric shapes. The substance is said to crystallise and each of the solid particles is called a crystal.

When the solution is not exactly saturated, *i.e.*, rather dilute, it may be heated till a thin pellicle of the salt begins to be formed on the surface of the liquid, removed from the source of heat and then allowed to crystallise slowly.

In this way prepare crystals of copper sulphate, alum, etc., and preserve them in sample tubes for the determination of their water of crystallisation (*vide* Chapter X).

DISTILLATION

It has been already shown that when ordinary impure water is boiled a solid residue is left behind. If the water vapours formed are not allowed to escape and are condensed by coming in contact with a cold surface we get what is called distilled water, and this

process consisting of evaporation and condensation is known as distillation.

Arrange an apparatus as shown in Fig. 24. Boil some water in the retort, which has been coloured blue by adding a few crystals of blue vitriol. The steam produced will pass into the receiving flask and con-

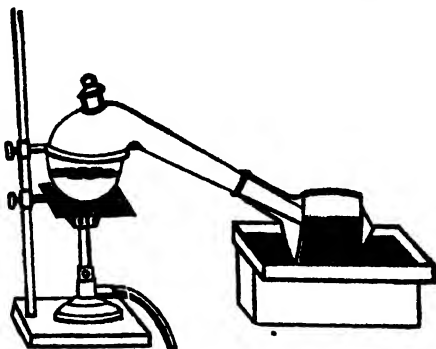


Fig 24

dense again. The flask is kept cool by being placed on a trough of water and covered with a piece of blotting paper or cloth moistened with water. Water collected in the receiver, known as distillate, will be colourless and free from the blue copper sulphate, formerly in solution.

Evaporate a little of the distilled water in a basin; it will leave no residue.

Next add some ammonia water to the blue solution in the retort and boil, observe that the distillate collected this time will smell strongly of ammonia. It is thus

evident that volatile impurities cannot be removed by distillation. Instead of the retort a distilling flask connected with a Liebig's condenser (Fig. 25) is conveniently used, especially in the case of the more volatile liquids like alcohol, ether, etc. In this case the

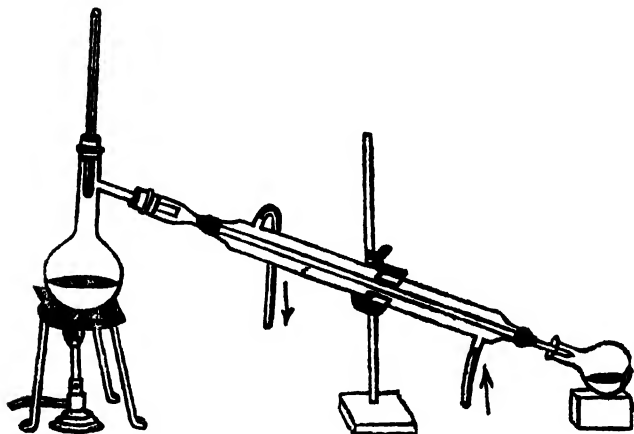


Fig. 25.

steam is condensed by passing through the inner tube surrounded by the jacket through which cold water is continually passed from a tap—the cold water entering the lower tubulure and hot water escaping from the upper one.

DETERMINATION OF THE BOILING POINT

The boiling point of water may be ascertained in the process of distillation with the help of a thermometer which should be inserted in the neck of distilling

flask through a bored cork. The bulb of the thermometer being above the surface of the liquid (Fig. 25), and only exposed to the vapours, it will indicate the boiling point of the pure liquid. If the bulb be dipped in the liquid itself, containing solids in solution, the boiling point will be raised. In this way determine the boiling points of solutions containing common salt, sodium sulphate, sugar, etc. Observe that the boiling point will be considerably above $100^{\circ}\text{C}.$, thereby proving that the boiling points of liquids are elevated by means of dissolved substances.

FRACTIONAL DISTILLATION

This process is commonly employed to separate two liquids boiling at different temperatures.

Take a distilling flask, provided with a thermometer to indicate the boiling point, as shown in Fig. 25. Connect the flask with a Liebig's condenser and introduce a mixture of water and alcohol in the flask. Apply heat and when the temperature reaches $78.3^{\circ}\text{C}.$ (the boiling point of alcohol) collect the first fraction of the distillate. Note that the boiling point of the liquid is gradually rising. Change the receiver when the thermometer registers 85° . In the same manner collect the distillate in three fractions up to $100^{\circ}\text{C}.$ which is the final temperature. Observe that the first portion of the distillate is very rich in alcohol and will burn when a flame is applied to it, while as the temperature approaches $100^{\circ}\text{C}.$ (boiling point of water) the proportion of water

increases and finally the distillate consists of water with very little of alcohol.

In this way two liquids of different boiling points may be separated from one another. The fractions may be separately redistilled and the substances obtained pure.

HOW TO DETERMINE THE SOLUBILITY OF A SUBSTANCE AT DIFFERENT TEMPERATURES

1. Take about 50 c. c. of water in a beaker, add some powdered potassium chlorate and stir with a glass rod. If the substance is completely dissolved add a little more of it until you get a saturated solution and some of the substance is left at the bottom. Observe that the temperature of the solution is slightly reduced. Wait till it comes to the temperature of the air. Note the temperature of the liquid and pour a few c.c. of the clear solution in a weighed basin and weigh the whole. Next evaporate the liquid in the basin to dryness on a water bath. When the residue is dry, wipe the bottom of the basin with a piece of cloth and leave it to cool in a desiccator. Weigh it when cold. Again heat it on the water bath, cool and weigh as before. Repeat the operations till the weight is constant. Calculate your results as follows.

(1)	Weight of basin	=	... grams
(2)	„ + solution	=	... „
			<hr/>

- | | | | | |
|--------------|-----------------------------------|---|-----------|---------|
| (3) | Weight of solution | = | ... | grams. |
| (4) | Weight of basin + KClO_3 | = | ... | " |
| | Weight of basin | = | ... | " |
| | | | <hr/> | |
| (5) | Weight of KClO_3 | = | ... | " |
| \therefore | Weight of water | = | (3) - (5) | = ... " |

From the above data calculate the amount of substance dissolved in 100 grams of water, which is the solubility of KClO_3 at the temperature of the room.

2. Now cool the beaker, containing the saturated solution prepared as before, by being surrounded with ice in a glass dish. After a few minutes the temperature falls nearly to zero. Note the temperature and pour carefully a little of the solution in a weighed basin and proceed as above. Whence calculate the solubility at that temperature.

3. Prepare a saturated solution of the substance at 50°C. This is done by placing the beaker in a water bath as shown in Fig. 26, which consists of a wide beaker containing water in which the one containing the solution is supported by means of corks. Now place the bath on a piece of asbestos board and heat by a small Bunsen flame. Regulate the flame so that the temperature of the solution remains stationary at 50°. When no more of the substance is dissolved, transfer a little of the solution, as rapidly as possible, into a stoppered weighing bottle of known weight. Allow



Fig. 26.

to cool and weigh again. Thus you get the weight of the solution. Now pour the solution, with any crystals that may have separated, into a weighed basin. Rinse the weighing bottle several times into the basin by means of a jet of water. Evaporate to dryness and weigh as before. Whence calculate the solubility at 50°C .

4. In the above manner, make a saturated solution at 100°C . and determine the solubility of the substance at that temperature.

From the above experiments trace the solubility curve of the substance in a squared paper making the

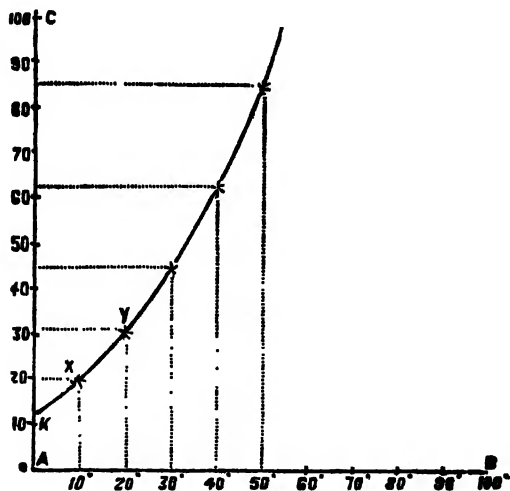


Fig. 27.

temperatures and the solubilities on abscissa and the ordinate respectively. Fig. 27 shows the solubility curve of nitre.

CHAPTER IV

CHEMISTRY OF FIRE

WHAT TAKES PLACE WHEN SOME SUBSTANCES ARE HEATED IN AIR

1. Take a clean porcelain crucible and weigh. Put a few bits of tin foil in the crucible and weigh again. Now support it on a pipe clay triangle placed on a tripod and heat it by a Bunsen flame (Fig. 28). After heating for a quarter of an hour, cool it and weigh again. You will find that the tin has increased in weight and has been converted into a grey substance which is called the *calx* of tin. *

2. Again take a few bits of shining copper turnings in a crucible and weigh. Heat it as before for half an hour; cool and weigh. An increase in weight has taken place. The copper no longer looks bright, but a black coating of calx of copper has been formed on the surface.

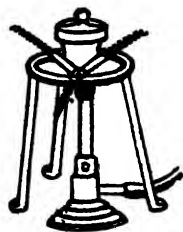


Fig. 28.

Thus many metals are converted into calces when heated in air, and the calces weigh more than the metal. The increase in weight is due to some constituent of air which the metals have taken up and is called the *active* part of air. Another part of the air which is left behind

* Cf. নব্য রসায়নী বিদ্যা ও তাঁহার উৎপত্তি, পৃষ্ঠা ১০—১৬

is called the inactive part, *i.e.*, substances will not burn in it as they do in air.

PREPARATION OF THE ACTIVE PART OF AIR

Take a little of the red calx of mercury in a hard glass test tube and heat it (Fig. 29). Collect the gas which is evolved in another test tube filled with water and

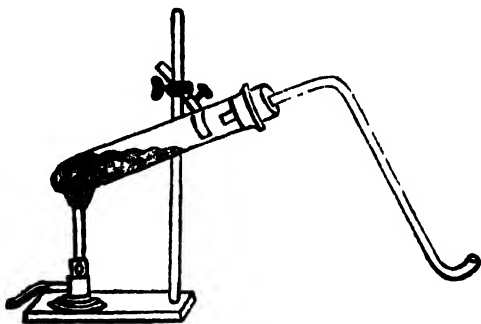


Fig. 29.

inverted over water in a pneumatic trough. Then introduce a glowing chip of wood into this test tube, it will at once burst into flame.

WHAT OCCURS WHEN A CANDLE BURNS

Have a bit of wax candle and light it. Leave it on the table for a quarter of an hour. Note the candle has diminished in size and hence in weight. What happens to the substance of the candle?

Fix the bit of candle in a candle holder. After lighting it, introduce it into a gas jar. Cover the mouth of the jar with a disc as shown in Fig. 30. The light will

now get more and more dim and after a minute or two will be extinguished. Take out the candle and pour a little clear lime water, cover the jar with a glass disc and shake up. The lime water has turned milky. Now introduce a lighted taper in the jar and you will see that it is extinguished. Pour a little clear lime water into another jar in which a candle has not been burnt. The lime water this time does not turn milky. Introduce a lighted taper which will burn in it as it does in air.



The burning of the candle has something to do with the milkiess of the lime water. Fig. 30. The substance of the candle in burning has taken up the active part of air forming an invisible gas called carbonic acid gas. Lime water takes up this gas and turns milky and what is left in the jar is only the inactive part of air.

CHAPTER V
**LAWS GOVERNING VOLUME AND
PRESSURE OF GASES**

BAROMETER

Take a glass tube about 36 inches long and half an inch in diameter and seal it at one end. After filling it with mercury close the open end with your thumb and

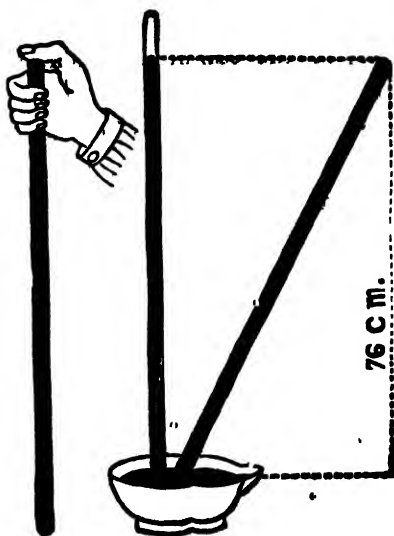


Fig. 31.

invert it in a trough containing mercury as shown in Fig. 31. Note that a column of mercury, about 30

inches or 76 cm. above the surface of the liquid in the trough is supported by the pressure of the atmosphere.

The space above the mercury in the tube does not contain any air as is proved by inclining the tube as shown in the figure, when the tube will be completely filled up with mercury.*

If the cross section of the tube were 1 sq. inch, the weight of the column of mercury would be about 15 lbs. In other words, the pressure exerted by the atmosphere is normally 15 lbs. per square inch.

The standard pressure for comparing gaseous volume is 760 millimetres.

VAPOUR PRESSURE OF A LIQUID

Fill a barometer tube with dry mercury and invert it in a trough of mercury as before. Surround the barometer tube by a wider glass tube or jacket as shown in Fig. 32. Through this jacket a current of steam can be passed from the top by boiling the water in the copper flask A. The steam escapes from the jacket by the tube B at its bottom.

Introduce a drop of water in the barometer tube by means of the pipette C. It will rise up and vaporise in the Torricellian vacuum and depress the column of mercury. On introducing more liquid drop by drop

* The space above the mercury is called the Torricellian vacuum, after the name of Torricelli, a pupil of Galileo, who first made this experiment in 1643.

the depression goes on increasing; but after a time the level of mercury remains constant. even if you go on introducing more water. It only goes up to form a

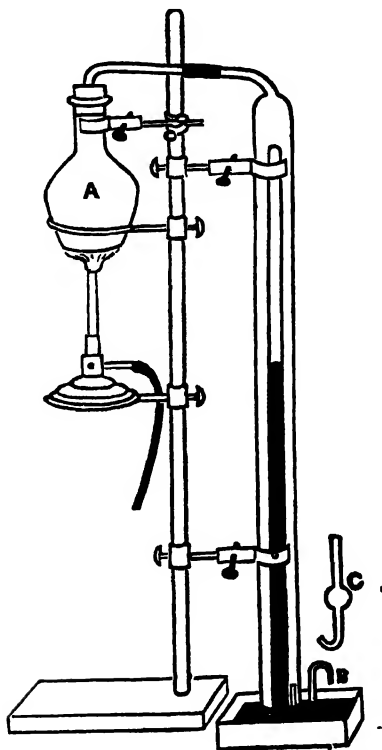


Fig. 32

layer over the level of the mercury The space above the mercury is now said to be saturated with water vapour.

This depression in the column of mercury is the tension of aqueous vapour at the temperature of the room.

Now half fill the flask with water and heat to boiling. As the steam passes through the outer jacket, more and more water is vaporised at the top of the barometer tube, and the mercury column is lowered, until the level of mercury in the tube is the same as in the trough.

At the boiling point of a liquid, its vapour pressure is equal to the atmospheric pressure.

Then stop the steam supply and leave the whole apparatus to cool down. See that the mercury rises up again in the tube. Measure the column of mercury. It is equal to the original height.

Now clean the tube and thoroughly dry the mercury and the inner walls of the tube so that there be no adhering moisture and fit it up again as before, but instead of water, introduce a few drops of ether. Note the height of the column of mercury at the temperature of the room and also after passing a stream of hot water (about 40°C .) through the outer jacket. The boiling point of ether is 35°C .*

BOYLE'S LAW

RELATION OF GAS VOLUMES TO PRESSURE

The influence of pressure on the volume occupied by a gas was first observed by Robert Boyle in the year 1661. It is as follows:—

* Ether is a very inflammable liquid. Experiments with it should not be done near a flame.

The volume occupied by a gas is inversely proportional to the pressure which it bears provided the temperature is constant.

If V is the volume of a gas under pressure P and V' the volume under pressure P' , the ratio would be

$$V : V' = P' : P.$$

$$\text{or } PV = P'V'$$

$$\text{or } PV = \text{a constant}$$

The truth of this statement can be verified by the student by means of the following apparatus. It consists of a graduated glass tube (Fig. 33) sealed at one end and the open end is joined to a spherical glass vessel by means of a thick walled rubber tubing and clamped to a long stand. The apparatus is filled with mercury which fills the whole of the india-rubber tubing and also a portion of the graduated tube and the bulb.

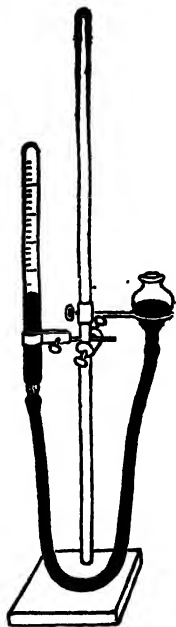


Fig. 33.

To perform the experiment, fix the closed tube near the base of the stand and adjust the position of the glass vessel so that the mercury stands at the same level in each. Read the volume of gas in the sealed tube. The pressure under which it stands is equal to that of the atmosphere, which is equal to the height of the barometer. Call this pressure P and the volume V . Now raise the bulb about half way up the stand and

note that the volume of gas in the sealed tube diminishes. Read this volume and call it V' . Measure the difference in the height of mercury in the two limbs and call it p . The pressure is now equal to this height plus the barometric pressure, *i. e.*, $P + p$.

Repeat the experiment several times at different pressures and see for yourself how far your results agree with Boyle's Law. Note that when the difference in the height of mercury in the two limbs is P , the volume is halved.

Here we have performed the experiment with increasing pressures. Now let us see if Boyle's Law holds good if we decrease the pressure.

To do this, fix the sealed tube at the top of the stand and adjust the level of the bulb. Read the volume of gas. The pressure is P . Lower the bulb half way down the stand and measure the difference in the level of mercury in the two limbs and call it p' . Read the volume in the sealed tube. The pressure is equal to $P - p'$.

Repeat the experiment several times with decreasing pressures and see whether PV is constant in each case.

What is the pressure when the volume of the gas is doubled?

EFFECT OF HEAT ON THE VOLUME OF A GAS

Fit up a flask of about 100 c. c. capacity with a good cork and a long capillary glass tube as in Fig. 34.

Take out the tube and suck in a little coloured liquid at the end nearer the cork. Fit it again. Now warm the flask by holding it between the hands. Note that the thread of liquid is pushed up, which is due to the expansion of the air within the flask. Again, let your hold go; the air contracts by cooling and the thread of liquid regains its original position



Fig. 34.

COEFFICIENT OF EXPANSION OF AIR

Take a round bottomed flask of about a litre capacity and fit it with a good india rubber cork, bored with one hole. Through this pass a piece of glass tube about 3 or 4 inches long, the outer end of which is fitted with a piece of indiarubber tubing which can be closed by a screw clip.

To begin with, see that the flask is perfectly dry. In order to be sure of that, pass a stream of hot air through the flask which will carry away all the moisture from inside the flask.

Now immerse the flask in a bath of water up to the cork and keep it in position by means of a clamp as shown in Fig. 35. Open the clip and heat the water in the bath. When the water has boiled for about a quarter of an hour, note the temperature of the water, which is 100°C . Remove the flame and screw the clip.

Now remove the flask and invert it in a bucket of old water and open the clip. Keep the flask immersed

in water for about five minutes, so that the air in it may cool down to the temperature of the water. Make the level of the water in the bucket the same as that in the flask and screw the clip. Note the temperature of

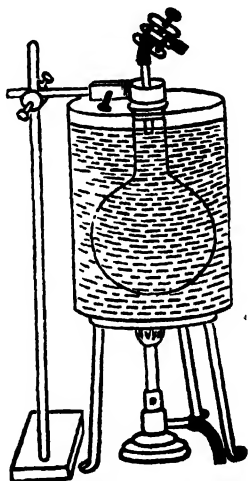


Fig. 35.

the water. Take out the flask and measure the volume of water which has entered. Again fill the flask with water up to the cork and measure the volume of this water, which is the capacity of the flask. Calculate your results as follows :—

Temperature of the boiling water = 100°C .

Temperature of water which entered the flask = 27.5°C .

Capacity of flask = 1075 c.c.

Volume of water which entered the flask on cooling from 100°C. to 72.5°C. = 209 c.c.

∴ Contraction of 1075 c.c. of air for (100 - 72.5) 72.5°C. = 209 c.c.

∴ Contraction of 1075 c.c. of air for 100°C.

$$= 209 \times \frac{100}{72.5}.$$

∴ 1075 c.c. of air at 100° will become

$$1075 - 209 \times \frac{100}{72.5} \text{ c.c. or } 786.72 \text{ c.c. at } 0^\circ\text{C.}$$

∴ Expansion of 786.72 c.c. of air for 100°

$$= 209 \times \frac{100}{72.5} \text{ c.c.}$$

or expansion of 1 c.c. of air for 100°

$$= \frac{209 \times 100}{72.5 \times 786.72}.$$

∴ Expansion of 1 c.c. of air for 1°C. or coefficient of expansion of air = $\frac{209}{72.5 \times 786.72} = \frac{1}{273}$ nearly.

Here you find that air expands by $\frac{1}{273}$ part of its volume measured at 0°C. for each increase of 1°C. And it has been found out by careful experiments that all other gases behave in the same manner, provided the pressure is constant. This generalisation is known as Charles' Law.

Suppose you have 273 c.c. of a gas at 0°C.

At $^{\circ}\text{C}$ the volume will be 274 c.c.

„ 2°C 275 c.c.

„ 50°C 323 c.c.

and so on

For temperatures below 0°C .,

273 c.c. at 0°C . will be 272 c.c. at ... 1°C .

... 271 c.c. „ 2°C .

... 223 c.c. „ „ 50°C .

and so on,

and theoretically at -273°C . the volume would be nil. This point has been termed the *absolute zero* of temperature. Hence 0°C . is equivalent to 273° of the absolute scale. Charles' Law may also be expressed in terms of the absolute scale, thus :

Under the same pressure, the volume of a gas varies directly as the absolute temperature.

The temperature of 0°C . has been taken as the standard for comparing gaseous volumes.

Normal temperature and pressure, or N. T. P., as frequently applied to a gas volume is 0°C . and 760 m.m. pressure.

Boyle's Law and Charles' Law can be combined in the form of a formula and which is frequently applied to calculate the volume of a gas at N. T. P. from the observed temperature and pressure.

Let V be the volume of a gas under pressure P and at temperature, $t^{\circ}\text{C}$.

Then the volume at N. T. P. will be,

$$V \times \frac{P}{760} \times \frac{273}{273+t}$$

DENSITY OF GASES

DENSITY OF OXYGEN

Take a dry round bottomed flask of about half a litre capacity, fitted with a rubber cork and two tubes which may be closed by two pinch cocks (fig. 36). Connect the longer tube with an oxygen reservoir and pass a slow current of the dried gas for about an hour so that the flask may be completely filled with oxygen. Close the pinch cocks and weigh the whole. Then exhaust the gas by a pump as far as possible. Close the pinch cocks and weigh again. Next invert the exhaust flask in a trough of water and open the cocks; water will rise in the flask. Close the cocks, take out the flask from the trough and pour the water which has entered into a measuring cylinder. Note the volume and temperature of this water. At the same time note the atmospheric pressure from the barometer. This volume of water indicates the amount of oxygen exhausted. The density of oxygen may be calculated as shown below from the results of an actual experiment.



Fig. 36.

Wt. of the flask + oxygen (before exhaustion)

= 255.751 grams.

Wt. of the flask + oxygen (after exhaustion)

= 254.634 grams.

Wt. of oxygen exhausted

= 1.117 grams

Volume of water which entered the flask = vol. of oxygen exhausted = 880 c.c.

Temperature = 25 C

Barometric height = 760 m m

Aqueous tension at 25 C. = 23.5 m. m.

Consequently, pressure = $760 - 23.5 = 736.5$ m. m.

Volume of the gas at N. T. P. (applying Boyle's and Charles' Laws) = 781.2 c.c.

∴ Weight of 781.2 c.c. of oxygen = 1.117 grams.

∴ Weight of 1 litre of oxygen = 1.43 grams.

Density = $\frac{\text{wt. of 1 litre of oxygen}}{\text{wt. of 1 litre of hydrogen}}$

$$= \frac{1.43}{0.09} = 15.89.$$

Theoretical density = 15.88.

DETERMINATION OF THE WEIGHT OF AIR

Take a flask fitted up as in the previous experiment ; dry it by passing a current of hot air and weigh with the pinch cocks closed. Then exhaust its air by the pump and weigh again. Next invert the exhausted flask over a trough of water and allow water to enter. Measure the volume of water which has entered the flask. This volume is equal to the volume of air exhausted. Note the temperature, the barometric height and also the aqueous tension at that temperature.

Suppose the volume of exhausted air, reduced to N. T. P. = v c.c.

Wt. of the flask + air = a (before exhaustion)

Density of Carbon Dioxide

Wt. of the flask (after exhaustion) = b

Wt. of air exhausted = $a - b$.

\therefore Wt. of 1 litre of air

$$= \frac{1000 (a - b)}{v} \text{ gram.}$$

The mass of a gas may be obtained by the volume which it occupies at N. T. P. multiplied by its density.

DENSITY OF CARBON DIOXIDE

Take the same flask fitted up as before and after drying it, take its weight. Then fill it completely with

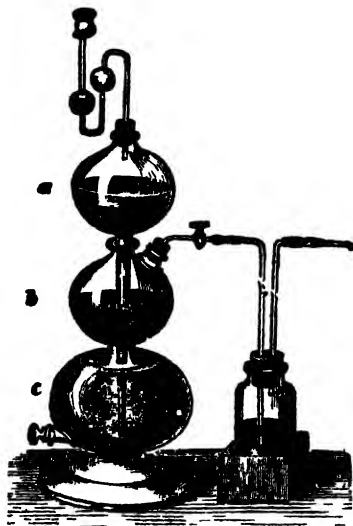


Fig. 37.

carbon dioxide by passing the dry gas from a Kipp's apparatus (Fig. 37). Weigh it again. Invert the flask

over a large dish containing some strong solution of caustic soda and open the pinch cocks. As carbon dioxide is absorbed by the caustic soda—the solution will rise and fill the flask completely. This is accelerated by gently agitating the flask. Measure the volume of the solution which enters the flask. This is equal to the volume of carbon dioxide. Note the temperature and barometric pressure. The tension of strong solution of caustic soda is negligible. Reduce the volume of the gas to N. T. P. and denote it by v .

Wt. of v c. c. of air = c (assuming the weight of 1 litre of air = 1.293 grams as calculated from the previous experiment).

Wt. of the flask + air = a .

Wt. of the flask + CO_2 = b .

\therefore Wt. of the vacuous flask = $a - c$.

Wt. of v c.c. of CO_2 = $b - (a - c)$.

Wt. of v c.c. of hydrogen at N.T.P. = $v \times 0.00009 \text{ gms.}$
(1 litre of hydrogen weighing 0.09 grams at N.T.P.).

\therefore Density of carbon dioxide

$$= \frac{\text{wt. of } v \text{ c. c. of the gas}}{\text{wt. of } v \text{ c. c. of hydrogen}}$$

$$= \frac{b - (a - c)}{v \times 0.00009} \therefore$$

•

CHAPTER VI

CHEMISTRY OF AIR—MIXTURES AND COMPOUNDS

COMPOSITION OF AIR

1. Fill a pneumatic trough with water to about half an inch above the shelf. Put a bit of phosphorus* in a small crucible floating on the water just above the shelf, and instantly cover the crucible by an inverted gas jar. The gas jar is previously divided into five equal marks along its length by a strip of paper as in Fig. 38.

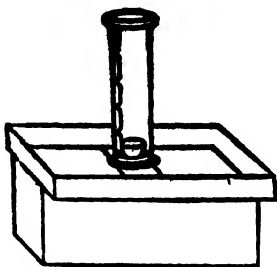


Fig. 38.

The phosphorus takes up the oxygen in the jar and the water rises up gradually to about one-fifth of the height of the cylinder, *i. e.*, up to the first mark.

* Owing to the danger incidental to the handling of phosphorus especially in the hot season, a beginner should not be allowed to perform this experiment, except in the presence of a demonstrator.

The residual air occupying four volumes is tested with a lighted taper, which is extinguished, proving it to be nitrogen which does not support combustion.

2 Make a small linen bag and fill it with clean iron filings. Take a gas jar, and a glass tube of the same length, and tie up the bag at one end of the glass tube by means of a string. Moisten the bag with water and introduce it into the jar inverted over water in the trough as shown in Fig. 39. The iron filings begin to rust at the expense of the oxygen in the jar. Leave the whole arrangement until the next day, when you will see that the water has risen up to one-fifth of the height in the jar. Test the residual air with a lighted taper.



Fig. 39.

Thus you see that air contains one-fifth of its volume of oxygen, which supports the combustion of all bodies and four-fifths of nitrogen which is an inert gas.

MIXTURES AND COMPOUNDS

IRON AND SULPHUR AND IRON SULPHIDE

Take about two parts by weight of iron filings and one part of sulphur and mix well by grinding them together in a mortar, and perform the following experiments.

(a) Take a little of the mixture on a piece of paper and pass a magnet over it. The particles of iron

will be seen to leave the mixture and stick to the magnet.

(b) Take a small portion of it in a test tube and pour dilute sulphuric acid over it. The acid attacks the iron only leaving the sulphur intact and evolving a gas having scarcely any odour.*

(c) Add a little carbon bisulphide to some of the mixture in a test tube and shake well ; the liquid will dissolve the sulphur leaving the iron particles undissolved. Filter and take a little of the filtrate in a watch glass or a small basin and place it over a water bath or simply blow with the mouth.* The liquid will evaporate off leaving yellow sulphur behind.

Now heat a little of the mixture in a hard glass test tube or in a clay crucible with the lid on over a Bunsen flame. The mixture begins to glow, finally becoming quite black. Observe also that a portion of the sulphur volatilises. The heating is now discontinued and the substance is allowed to cool, taken off the test tube and powdered. Repeat experiments (a), (b) and (c) with this powder. It may be slightly attracted by the magnet, if there is any free iron left. Carbon bisulphide will not

* As ordinary iron filings contain traces of carbon chemically combined with it (as carbide of iron), the hydrogen evolved is not absolutely odourless. See under "Carbide of iron and Acetylene." *Inorganic Chemistry* pp. 172 and 189.

* Carbon bisulphide is a very inflammable liquid. All experiments with it should be performed far away from any flame.

dissolve any sulphur from it and when treated with dilute sulphuric acid, it will give off a gas having the odour of rotten eggs and which will blacken a piece of paper soaked in lead acetate solution.

This change of properties is due to the fact that the iron and sulphur in the mixture have combined together under the action of heat to form a chemical compound, *iron sulphide*.

CHAPTER VII

STUDY OF THE PROPERTIES OF SOME ACIDS AND ALKALIES

Take some vinegar in a test tube and note its sour taste. Pour a few drops of it on a piece of blue litmus paper which will turn red, proving the acid property of the substance. Compare its smell with that of dilute acetic acid. Pour some vinegar on a small piece of chalk and a bit of granulated zinc placed in separate watch glasses and observe the action.

Take some strong sulphuric acid in another test tube and observe its oily appearance. Add very carefully a few drops of this acid, to about an ounce of water in a beaker. Stir with a glass rod and observe the heat evolved. Note also the sour taste of this diluted acid. Add some of this dilute acid to some chalk powder and to some granulated zinc in separate test tubes and notice that effervescence is set up in each case due to the evolution of gases. Observe its action on blue litmus which will be turned red.

N. B.—The student should be very careful at the time of using strong acids which are highly corrosive substances and may produce dangerous ulcers and destroy clothes if carelessly handled.

Perform similar experiments with hydrochloric and nitric acids separately and note (1) that fumes are evolved

when the bottles of these acids are opened; (2) that hydrochloric acid added to zinc granules also produce an invisible gas with effervescence while nitric acid produces red fumes; (3) that they also redden blue litmus.

Take the solutions, obtained by the action of sulphuric and hydrochloric acids on zinc. Filter and concentrate the clear liquids by boiling them down in separate basins. Set them aside to cool. Needle-shaped crystals will appear in one and no crystals in the other.

Also observe that by the action of sulphuric acid upon chalk an insoluble white substance is produced and that by the action of hydrochloric acid, the chalk disappears and a clear solution is obtained.

Next take some bits of caustic soda and caustic potash and expose them to air in separate watch glasses and observe that they become moist and ultimately liquefy. Add some water to them in separate basins. They are seen to dissolve. Dip your fingers in the solutions thus obtained. Observe the rise of temperature of the liquids and notice the soapy feel they impart to the skin. Add one or two drops of the solutions to red litmus papers, which will turn blue, showing their alkaline properties.

Take some clear lime water and test with a red litmus paper. It will also turn blue. Taste a few drops of it and note its caustic taste.

Substances such as caustic soda or potash or lime are, therefore, known as alkalis in contradistinction to

the class of bodies examined before and known as acids. When these two classes of bodies, *viz.*, acids and alkalies, are brought together they produce new substances, called salts, in which neither the acid nor the alkaline properties of the original substances exist. They will be discussed fully later on (*vide* Chapter on Acidimetry and Alkalimetry).

CHAPTER VIII

OXYGEN

1. Take some crystals of potassium chlorate in a hard glass test tube and apply heat. At first, slight crackling sounds are heard, then the salt melts and at last gives off a gas. Test with a glowing chip of wood ; it is rekindled. The potassium chlorate decomposes at a high temperature giving off oxygen.

2. Mix some potassium chlorate with about one fourth of its weight of manganesé dioxide; half fill a hard glass test tube with this mixture and fit it up with a cork and delivery tube as shown in Fig. 29. Now carefully apply heat ; the gas will be given off at a lower temperature than if only potassium chlorate were used. Unless the heat is carefully regulated in this case, the evolution of the gas may be so rapid as to produce an explosion.

Collect several jars of the gas by the displacement of water over a pneumatic trough and apply the following tests.

1. Insert a glowing chip of wood into a jar of the gas ; it is at once rekindled and bursts into flame.

2. Introduce a piece of glowing charcoal into a jar or bottle of the gas, placing the charcoal in a deflagrating spoon (Fig. 40). Notice that it burns with dazzling

brilliancy Now pour a little clear lime water into the bottle ; it is turned milky



Fig. 40



Fig. 41.

3. In the same manner burn a piece of sulphur in another jar ; it burns with increased brilliancy. Pour a little blue litmus solution ; it is turned red.

4. Burn a bundle of iron wire or a steel watch spring in a jar or bottle of oxygen ; the lower end of the iron wire being previously tipped with molten sulphur and ignited. Observe that the iron burns with showers of sparks (Fig. 41).

N. B. The bottom of the glass jar should be covered with a layer of sand.

5. Cut off a piece of phosphorus of the size of a pea under water in a basin. Quickly dry it by pressing between folds of a piece of blotting paper and place it on a deflagrating spoon. As phosphorus is highly

combustible it should be ignited by touching with a heated metallic wire but never in a direct flame*.

Introduce the ignited phosphorus into a jar of the gas ; it burns with dazzling light forming white clouds. Pour a little blue litmus solution ; it is turned red.

RECOVERY OF POTASSIUM CHLORIDE

When the evolution of oxygen has ceased, set aside the test tube to cool. Then dissolve out the contents with water and filter. To a little of the solution in a test tube add a drop of silver nitrate solution ; a white curdy precipitate is formed. Observe the difference by adding another drop of silver nitrate solution to a little potassium chlorate solution when there will be no precipitate.

Now evaporate the filtrate in a basin and obtain crystals of potassium chloride.

* This experiment with phosphorus should be done only in the presence of the demonstrator.

CHAPTER IX

HYDROGEN

Take a two-necked Woulff's bottle fitted with a thistle funnel and a delivery tube as shown in Fig. 42. Place some granulated zinc in the bottle and cover with a layer of water. Pour some dilute sulphuric acid through the funnel; immediately a brisk evolution of gas takes place. Wait for two or three minutes to allow all the air to escape. Collect samples of the gas in test tubes and apply light. When the gas burns quietly without any report collect several jars of the gas over water in a pneumatic trough and perform the following experiments:

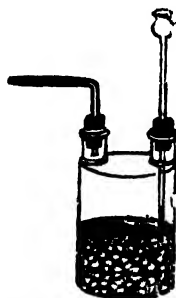


Fig. 42

1. Hold a jar of the gas mouth downwards and thrust a burning taper into it; the gas burns at the mouth but the taper is extinguished.

2. To see that the gas is lighter than air pour it upwards into another counterpoised jar as in Fig. 43, and test with a lighted taper. Observe that the gas burns with a slight explosion in the upper jar, while, the lower one, originally full of hydrogen, will be practically free from hydrogen and will not burn.

Connect the delivery tube of your apparatus with a straight calcium chloride tube ending in a jet as shown

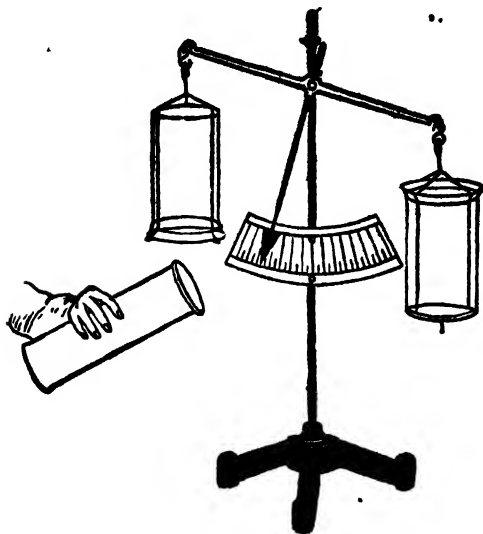


Fig. 43

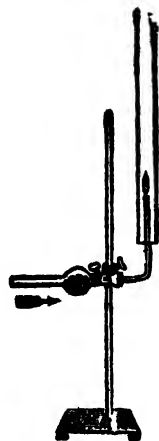


Fig. 44.

in Fig 44. Apply a flame to the issuing gas and surround the burning jet by a long wide glass tube open at both ends. Observe that drops of water condense in the cooler parts of the tube. If the tube be pushed up and down very cautiously a point will be reached when a singing or even a moaning note will be produced.

RECOVERY OF CRYSTALS OF ZINC SULPHATE

Take the liquid left in the hydrogen generating bottle and filter. Evaporate the clear filtrate in a basin

till a thin white crust is just visible on the surface of the liquid. Allow to cool ; crystals of zinc sulphate, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$, will appear after some time. Take out the crystals from the mother liquor, redissolve them in water and crystallise again. Fish out the crystals, dry them between folds of blotting paper and preserve them in a sample tube

CHAPTER X

WATER

TO PROVE THE VOLUMETRIC COMPOSITION OF WATER BY ELECTROLYSIS

Take an ordinary voltameter as shown in Fig. 45, which can be easily prepared in the laboratory. Half fill it with distilled water. Then take two equal graduated test tubes, fill them with water, close their open ends with your thumb and invert them over the two platinum strips, called electrodes. Connect the two electrodes with the two terminals of an electric battery, when no action will be observed as pure water is a non-conductor of electricity. Next add about 1 c. c. of sulphuric acid and mix it with the water by stirring with a glass rod. Notice that bubbles of gases are now liberated from the two platinum electrodes and that the rates of evolution are also unequal. Collect the gases in the two test tubes. After some time disconnect the battery and remove the test tubes, closing their open ends with the thumb, and invert them over water in two jars. On

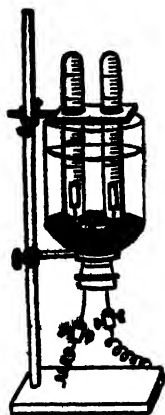


Fig. 45.

adjusting the levels of water both inside and outside the two tubes, read the volume of the gases and notice that the volume of the gas in one tube is nearly double that in the other. On testing with a glowing chip of wood, the gas which occupies the smaller volume is proved to be oxygen and the other gas which occupies the larger volume is seen to be hydrogen as it takes fire with a slight explosion and burns with a non-luminous flame when a lighted taper is presented to it. Thus, you prove that water is made up of two volumes of hydrogen and one volume of oxygen.

GRAVIMETRIC COMPOSITION OF WATER BY SYNTHESIS

To determine the weights of hydrogen and oxygen which combine to form water, arrange an apparatus as shown in Fig. 46. Take a hard glass tube about one foot long through which a porcelain boat can be freely

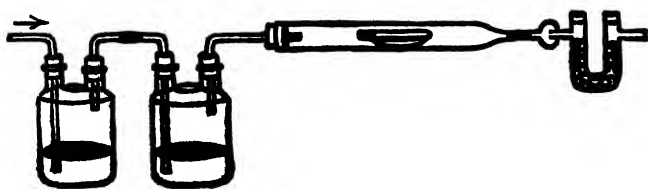


Fig. 46.

passed, One end of the tube should be closed with a cork through which passes a short narrow glass tube and the other end should be drawn out. Hold the tube in a horizontal position by means of clamps. Take

about one gram of black copper oxide * in the porcelain boat. Carefully weigh the boat with the copper oxide and introduce it into the central part of the tube by means of a wire. Connect the end of the tube, fitted up with the cork and the narrow glass tube, with a Kipp's apparatus for the supply of hydrogen which should be purified and dried by being successively passed through two gas bottles containing potassium permanganate solution and strong sulphuric acid and join the drawn out end with a calcium chloride U tube, provided with a moisture trap, the weight of which should be determined. The U tube is filled up with fused and granulated calcium chloride, which has great attraction for water. The limbs are closed with good corks and the side tubes are closed with pieces of indiarubber tubes and stoppers made of glass rod which excludes the external air when the tube is not in use.

Pass a current of dried hydrogen through the tube and heat the copper oxide strongly for about half an hour, taking care that no moisture is condensed at the end of the tube, which should also be heated now and then by means of a separate burner. All the water produced by the union of the hydrogen with the oxygen of the copper oxide is driven into the drying U tube where the portion condensed is collected in the side bulb and the rest is absorbed by the calcium

* The copper oxide should be previously dried by being heated in a crucible and cooled in a desiccator.

chloride. Observe that the black copper oxide has become red owing to its reduction to metallic copper. Allow the copper to cool in the current of hydrogen. When the tube has cooled, take out the porcelain boat gently by means of a hooked copper wire and weigh. Again place it in the tube and heat it as before for another ten minutes. Cool and weigh again. Repeat this process till no further loss of weight occurs. Weigh also the calcium chloride tube after the experiment is over. Enter your results thus :

- | | | | |
|-----|---|---|------------|
| (1) | Wt. of the boat + CuO | = | ... grams. |
| (2) | „ „ + copper | = | ... grams. |
| (3) | „ of oxygen (by difference) | = | ... grams. |
| (4) | „ of Ca Cl ₂ tube before ex- | | |
| | periment | = | ... grams. |
| (5) | „ after experiment | = | .. grams. |
| (6) | „ of water formed (difference | | |
| | between (4) and (5).) | = | ... grams. |
| | Wt. of hydrogen (difference | | |
| | between (6) and (3).) | = | ... grams. |

Calculate the combining proportions of hydrogen and oxygen to form water. Theoretically, 8 grams of oxygen combine with 1 gram of hydrogen to form 9 grams of water. From these calculations the equivalent of oxygen is also found to be 8.

WATER OF CRYSTALLISATION

Heat a few crystals of copper sulphate in a dry hard glass test tube and notice that the dry crystals give off a

certain quantity of water which are condensed in the cooler parts of the tube. Also observe that at the first heating, crackling sounds are heard owing to the bursting of the crystals by the sudden expansion of the mother liquor enclosed within. Continue heating until no more water is expelled and notice that the crystals have lost their geometric forms and are changed into a white amorphous powder. This water is known as the *water of crystallisation*. It may be said to form part of the crystals and chemically combined with the substance rather feebly.

Note also that when a drop of water is added to the anhydrous copper sulphate, it slowly regains its original blue and crystalline form.

DETERMINATION OF THE WATER OF CRYSTALLISATION OF COPPER SULPHATE CRYSTALS

In order to determine the amount of water of crystallisation accurately, take some powdered crystals of copper sulphate. Weigh a clean, dry porcelain crucible with its lid and introduce into it about a gram of the substance and weigh again. Heat the crucible with its contents and lid on in a hot-air bath raised to about 110° to 115°C. , which is indicated by a thermometer. After half an hour take it out from the air bath, cool in a desiccator (Fig. 47) and weigh. Heat it again in the same bath for about twenty minutes, allow to cool and take its weight. If any further loss in weight is observed, the operation should be repeated until the weight is constant. In this

way the copper sulphate crystals will be deprived of only four molecules of water of crystallisation. In order to remove the last, *i. e.*, the fifth molecule of water, the crucible with its contents and lid will have to be directly heated very cautiously by a small Bunsen flame, which will just touch the bottom of the crucible or the crucible may be placed on an asbestos board and gently heated by a Bunsen flame avoiding high temperature.



Fig 47.

For very accurate determinations the crucible might be heated in an air-bath raised to about 240°C .

After heating for some time cool it in a desiccator and weigh. Repeat the process until you get a constant weight. Enter your results as follows:

- | | | |
|--|---|------------|
| (1) Wt. of crucible with lid | = | ... grams. |
| (2) Wt. of crucible with lid + substance | = | ... grams. |
| (3) Wt. of substance taken (by difference) | = | ... grams. |
| (4) Wt. of crucible with lid + substance (after heating) | = | ... grams. |
| (5) Wt. of water lost (subtracting (4) from (2).) | = | ... grams. |

Whence calculate the percentage of water in the crystals in the two stages.

Theoretically, copper sulphate crystals, $\text{Cu SO}_4 \cdot 5\text{H}_2\text{O}$, contain about 36.14 per cent of water of crystallisation.

The student should also try to determine the water of crystallisation of zinc sulphate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) and of potash alum ($\text{K}_2 \text{SO}_4 \cdot \text{Al}_2 (\text{SO}_4)_3 \cdot 24\text{H}_2\text{O}$).

Note. Potassium nitrate, common salt and many other salts do not contain water of crystallisation.

CHAPTER XI

NITROGEN AND ITS COMPOUNDS

NITROGEN

Take a flask of about a litre capacity, pour into the flask a small quantity of a concentrated solution of a mixture of ammonium chloride and sodium nitrite, which by double decomposition yield ammonium nitrite. Close the neck with a good cork carrying a thistle funnel and a delivery tube (Fig. 48), the end of which should dip

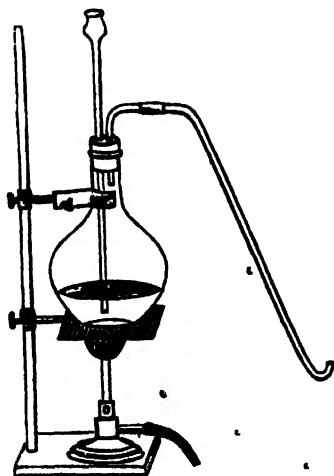


Fig. 48.

under water in a pneumatic trough. Heat the flask carefully by a rose burner. As the liquid froths up greatly owing to the brisk evolution of the gas, the heating

should be properly regulated. As soon as the gas begins to evolve, remove the source of heat. Collect the nitrogen, which is liberated, in some jars by the displacement of water over the pneumatic trough and apply the following tests.

(1) Insert a burning taper into one of the jars full of the gas. It is at once extinguished and the gas does not take fire.

(2) Pour some clear lime water into another jar. It is not turned milky.

NITRIC ACID

Introduce about 25 grams of nitre into a small stoppered retort (see Fig. 24) the neck of which passes into a small flask which is called the receiver. Pour some strong sulphuric acid through a funnel taking care that no acid runs down the long neck of the retort. Replace the stopper and heat the retort gently by means of a Bunsen burner with a rose top. After a short time, the nitric acid begins to distil over and condense as a yellowish fuming liquid in the flask kept cool by being placed in a trough of water and covered with a piece of blotting paper or cloth, which is also moistened with cold water.

Pour out the hot residue of potassium hydrogen sulphate left in the retort, otherwise it will solidify into cake on being cooled and cannot be easily removed afterwards. Now apply the following tests :

✓ (1) Add a few drops of the acid to some copper turnings in a test tube and notice the red fumes that are evolved and the blue solution of copper nitrate left in the test tube.

✓ (2) Add a few drops of the acid to some granulated zinc in another test tube and notice that reddish fumes are evolved. In this case also the colourless hydrogen gas is not liberated.

(3) Add a few drops to granulated tin. Red fumes are also given off and a white residue is left.

✓ (4) Observe the action of nitric acid on indigo carmine solution the colour of which is turned brown, and on blue litmus which becomes red.

✓ (5) *Ring test of nitric acid*:—Take a drop or two of the acid in a test tube and mix with a little strong sulphuric acid and allow the mixture to cool. Now pour cautiously down the side of the test tube, which is held somewhat slantingly, a small quantity of freshly prepared solution of ferrous sulphate. A dark brown ring will appear at the zone of contact of the two liquids. This ring is unstable and disappears on shaking or on warming. At the time of making the ferrous sulphate solution, care should be taken not to use crystals which have turned yellow. The yellow incrustation should first be removed by repeated washing with distilled water and the solution is to be made of green crystals alone.

Note—All experiments with nitric acid should be performed in a draught chamber, if possible, as the red fumes evolved are poisonous and should not be inhaled.

NITROUS OXIDE OR LAUGHING GAS

Take a flask of about 500 c. c. capacity and place in it a quantity of dry ammonium nitrate crystals. Close the neck with a cork carrying a delivery tube (Fig. 40). Heat the substance until it melts and gives off bubbles of gas which should be collected in jars by the dis-

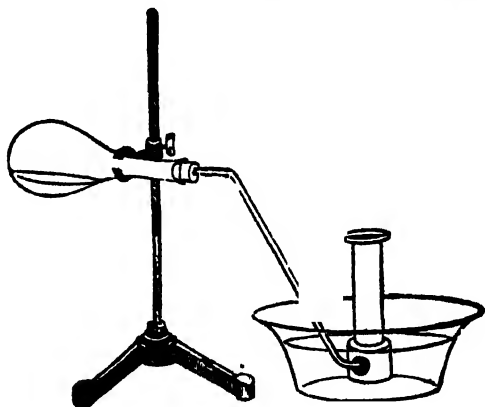


Fig. 49.

placement of hot water over a pneumatic trough. The substance should not be heated too strongly as in that case it will decompose with explosive violence. Collect several jars of the gas and apply the following tests.

(1) Introduce a glowing chip of wood into one of the jars. It bursts into flame and the gas does not take fire.

(2) Hold a jar inverted over cold water in a trough for some time. The water is seen to rise slowly in the jar.

(3) Remove the disc from the mouth of one of the jars. The gas produces no red fumes on coming in contact with air.

(4) Ignite a small bit of sulphur in a deflagrating spoon and when the sulphur is burning feebly, introduce it into one of the jars when the sulphur is extinguished. Again allow the sulphur to burn brightly in air and then plunge it into the jar. It will continue to burn with increased brilliancy.

(5) Take a small bit of phosphorus of the size of a pea in a deflagrating spoon. Light it with a wire heated in the Bunsen flame and plunge it into a jar full of the gas when the phosphorus is seen to burn with dazzling brilliancy and dense white fumes fill the jar.

N. B. The gas as ordinarily prepared is not pure and the student must not inhale it to prove its property of inducing laughter

NITRIC OXIDE

Place some copper turnings in a Woulff's bottle fitted with a thistle funnel and a delivery tube as in Fig. 42. Add a little water to cover the turnings. Pour some nitric acid down the thistle funnel. Reddish brown fumes are at once seen to fill the bottle and the solution becomes greenish blue owing to the formation of copper nitrate. Allow the gas to escape for some time and when the atmosphere inside the bottle has become almost colourless, begin to collect the gas in several jars by displacement of cold water over a pneumatic trough.

Cover the mouths of the gas jars with greased glass discs. Perform the following experiments with the gas.

(1) Remove the disc from the mouth of one of the jars and notice that red fumes are formed when the colourless gas comes in contact with the atmospheric oxygen. Add a little water to the jar and shake up; the fumes are dissolved and a solution is produced which turns blue litmus red.

(2) Introduce a lighted splinter into another jar. It is extinguished and the gas does not take fire.

(3) Introduce a bit of burning sulphur by means of a deflagrating spoon into another jar; it is extinguished.

(4) Introduce a piece of feebly burning phosphorus into a jar—it is also extinguished; but on taking out the phosphorus and allowing it to burn brightly in air, plunge it again into the jar when it is seen to burn still more brightly and produce copious white fumes.

(5) Close the mouth of a test tube full of the gas with your thumb. Invert it and open the mouth under the surface of a strong ferrous sulphate solution, when the solution becomes brown and rises up in the test tube.

(6) Add two or three drops of carbon bisulphide by means of a pipette into a jar full of the gas by slightly opening the mouth. Close it and shake up, when the vapour gets mixed with the gas. Now hold the mouth before a gas flame when the mixture burns off with a brilliant blue flash.

AMMONIA

Take some ammonium chloride and notice that it has no odour. Rub a little of this substance mixed with powdered quicklime in a mortar and notice the pungent smell that is given off. Introduce some of the mixture of lime and ammonium chloride into a round bottomed flask and place some bits of quicklime above the mixture. Close the mouth with a cork carrying a bent tube and arrange it as shown in Fig. 50. Heat the flask

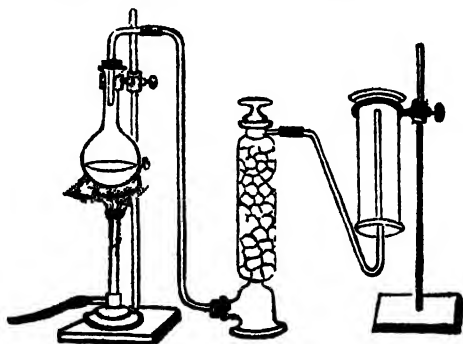


Fig. 50.

gently by a Bunsen flame. Collect several jars of the gas by the upward displacement of air as shown in the figure, and perform the following experiments.

(1) To show that ammonia is lighter than air, pour ammonia upwards from one test tube into another and prove its presence in the upper test tube by plunging in it a piece of moist red litmus paper which will turn blue.

(2) Introduce a lighted taper into a jar of the gas ; it will be extinguished but the gas appears to burn with a greenish yellow flame when it meets the lighted taper. The flame of burning ammonia can be seen by holding the end of the delivery tube just at the tip of the Bunsen flame when a flickering yellow flame will appear.

(3) Hold a glass rod dipped in hydrochloric acid in a jar of ammonia when white fumes will be at once formed. This experiment may conveniently be shown by taking two similar jars full of ammonia and hydrochloric acid respectively and bringing them mouth to mouth when dense white fumes will fill the two jars.

(4) To prove the extreme solubility and the alkalinity of ammonia, take a jar full of the gas with its mouth closed with a disc. Invert it over water, coloured with red litmus in a trough. Push aside the disc slightly so as to bring the gas in contact with water when the water rushes in and fills the jar and the colour of the liquid becomes blue.

1) B. "The 'Ammonia' experiment. (Vide book, A 83)



CHAPTER XII .
CHLORINE AND ITS COMPOUNDS
HYDROCHLORIC ACID

Take some common salt in a flask (Fig. 51) and add sulphuric acid, previously diluted with an equal

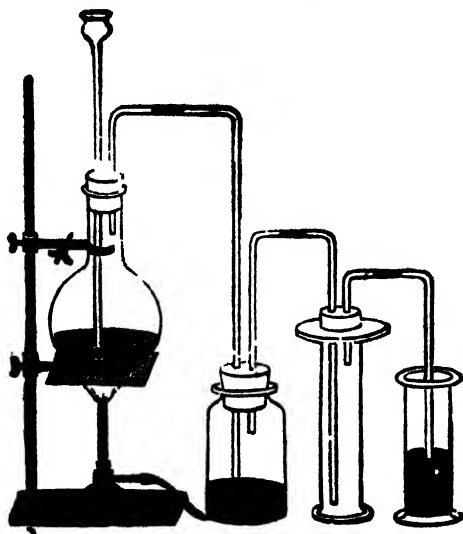


Fig. 51.

volume of water,* through the thistle funnel. Warm the mixture and pass the gaseous hydrochloric acid

* For this experiment about 50 c.c. of strong sulphuric acid may be cautiously added in a thin stream into an equal bulk of water in a basin and stirred with a glass rod. The basin should be placed in a vessel of cold water for keeping it cool.

through strong sulphuric acid in a wash bottle. Collect the dry gas in some jars by downward displacement. cover with glass plates and perform the following experiments.

(1) Remove the glass plate from one of the jars and notice that the gas fumes in contact with moist air.

(2) Hold a glass rod moistened with ammonia solution near the mouth of another jar; dense white clouds of ammonium chloride are formed.

(3) Test the gas with a burning taper, which will be extinguished and the gas will not burn.

(4) Invert a jar of the gas, mouth downwards, in a basin of water which has been coloured blue by the addition of litmus and observe that the water rapidly rises and is also turned red showing the extreme solubility of the gas and also its acid character.

(5) Saturate a small quantity of distilled water with the gas in a test tube and test the aqueous solution with the following substances in separate test tubes.

(a). a few granules of zinc; observe the effervescence.

(b) a few drops of silver nitrate solution, when a white curdy precipitate will be obtained; this precipitate is soluble in ammonium hydrate and insoluble in nitric acid.

CHLORINE

Introduce some manganese dioxide into a flask, as shown in Fig. 51, and pour strong hydrochloric acid.

Warm the mixture and collect 'a few jars of the gas by the downward displacement of air, after passing it through the wash bottle containing water.

(1) Allow the gas to bubble through some distilled water in a beaker and notice its solubility. Keep this solution, known as "chlorine water", in a stoppered bottle.

V. B. As chlorine is *highly poisonous* it *should not be inhaled* and should always be prepared in a stink cupboard with a flue.

(2) Insert a lighted taper in a jar of chlorine and observe that it burns with a lurid smoky flame, evolving fumes of hydrochloric acid and liberating carbon in the form of soot; the gas itself does not burn.

(3) Throw some powdered antimony and a leaf of Dutch metal successively into two jars and notice the formation of the chlorides of the metals with the evolution of light and heat.

(4) Introduce a piece of turkey red cloth, a strip of litmus paper and a red flower respectively moistened with water in jars of the gas and note that they are all bleached.

(5) Take a piece of printed paper dipped in ordinary writing ink and expose it to the action of chlorine; observe that the ordinary ink only is bleached, while the printed letters remain unchanged.

Perform experiment (4) with 'dry chlorine and notice the difference.

CHAPTER XIII

OXIDES OF CARBON

CARBON DIOXIDE

(1) Heat some calcium carbonate (chalk powder) in a hard glass test tube, closed with a cork and fitted with a delivery tube, and pass the evolved gas into clear lime water (Fig. 52). Observe that the lime water will be turned milky owing to the evolution of carbon dioxide.

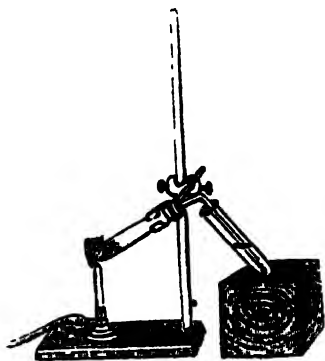


Fig. 52.

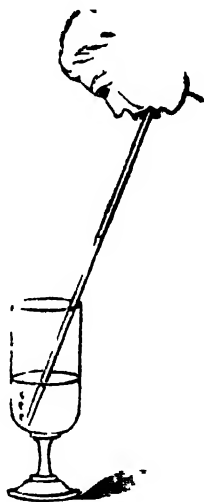


Fig. 53

(2) Blow from your mouth into clear lime water in a test glass or beaker and observe its turbidity—proving

that carbon dioxide is formed during the process of respiration (Fig. 53).

(3) Place some chips of marble in an ordinary Woulff's bottle (see Fig. 42) ; cover them with water and pour hydrochloric acid through the thistle funnel. Notice the brisk effervescence. Collect the gas in some jars by the downward displacement of air.

(i) Insert a burning taper in a jar and observe that the gas does not take fire, while the taper is at once extinguished.

(ii) Prove that the gas is heavier than air by pouring it (like water) from one test tube into another full of air. Notice that a lighted taper will be extinguished in the lower test tube which now contains carbon dioxide by displacement of air.

(iii) Bubble the gas through some blue litmus solution and observe that the colour is changed to *port wine red*. Boil this red liquid and notice that the blue colour is restored proving the feeble acid character of carbonic acid. Compare this reaction with the reddening of litmus by sulphuric acid.

(iv) Pass a stream of the gas through some clear lime water largely diluted and observe that a turbidity is first produced, which again disappears and the liquid becomes clear on the continued passing of the gas. Pour the clear solution of calcium bicarbonate thus formed into two beakers. Test one part with soap ; no lather will be at first formed, proving the solution to be

hard water. Boil the second portion for some time, the turbidity will reappear. Filter off the precipitate and test the clear filtrate again with soap solution, which will produce a lather easily, showing that the water has been softened by boiling.

AMOUNT OF CARBON DIOXIDE IN A CARBONATE

Take a small flask of about 200 c.c. capacity, and fit it up with a good cork bored with two holes. Through one pass a bent glass tube and through the other a straight calcium chloride tube filled with this substance and plugged with cotton wool, if necessary, as shown in Fig. 54.



Fig 54.

Before closing the flask, put a little water in it and introduce a small test tube half filled with hydrochloric acid and suspend it from the cork by means of a thread. Take care that no acid may drop out of the test tube into the flask. Carefully weigh the whole arrangement.

Now slowly take out the cork with the test tube and drop a small piece of marble into the flask. Replace the cork and test tube. Weigh again.

Then tilt the flask so that a portion of the acid runs out into the flask. Immediately action begins with effervescence and carbon dioxide is evolved. Wait till all the marble is dissolved and the liquid appears quite clear. The space above the surface of the liquid is now

of course filled with carbon dioxide, and in order that we may know the weight of the marble minus carbon dioxide, this must be driven out of the flask. This is done by sucking through the calcium chloride tube so that air may enter through the bent tube and bubble through the liquid.

Now weigh and calculate your results as follows

First weight	...	=	... grams.
Second weight	.	=	... "
Difference (weight of marble)		=	. "
Second weight	..	=	... "
Third weight	.	=	.. "
Difference (weight of carbon dioxide)	...	=	. "

Percentage of carbon dioxide =

Theoretical percentage of carbon dioxide in marble is 44.

CARBON MONOXIDE

Arrange an apparatus as shown in Fig. 55, and introduce about ten grams of oxalic acid into the flask and cover it with strong sulphuric acid. The first two Woulff's bottles contain strong solution of caustic soda in order to absorb the carbon dioxide which will also be evolved along with the carbon monoxide in the reaction. The third contains lime water which is placed to see if the gas is free from carbon dioxide. Heat the contents of the flask gently, and when the air has been expelled, collect a few jars of the gas by the displacement of

water over a pneumatic trough and cover them with glass plates.

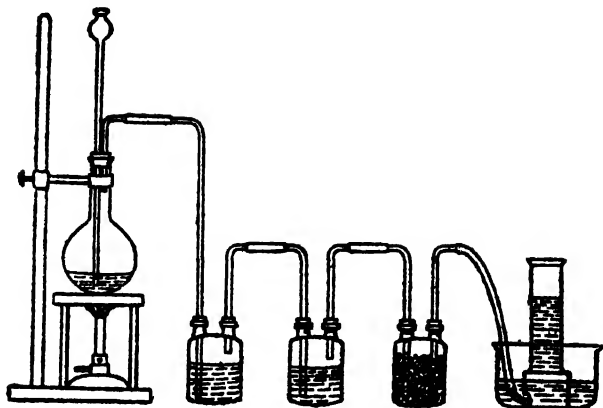


Fig. 55.

(1) Remove the glass plate from one of the jars and introduce a lighted taper; observe that the colourless gas takes fire and burns with a pale blue, lambent flame, while the taper is extinguished. Pour lime water into the jar when the combustion is finished; it is turned milky proving that carbon dioxide is the product of burning carbon monoxide in air or oxygen.

(2) Pour a little lime water into another jar of carbon monoxide; it is not turned milky.

N. B. Carbon monoxide is a highly poisonous gas. It should be always burnt when produced and never inhaled or allowed to escape in the air. It should be prepared in a stink cupboard provided with a flue.

CHAPTER XIV

SULPHUR AND ITS COMPOUNDS

ALLOTROPIC FORMS OF SULPHUR

(1) *Rhombic Sulphur*

Take some powdered sulphur in a test tube and pour a few c.c. of carbon bisulphide into it. Shake well to dissolve the sulphur. Filter and evaporate the filtrate on a clock glass either on a water bath or by simply blowing with the mouth. In a short time you will notice that small shining yellow crystals have separated out. They are distinctly seen by the aid of a lens to be "octahedra," belonging to the rhombic system (Fig. 56. A). This is the most stable variety and it is soluble in carbon bisulphide

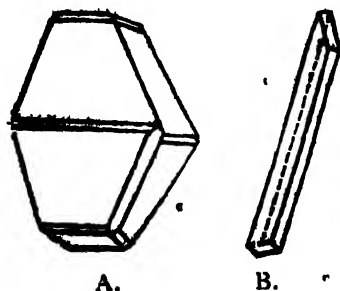


Fig. 56.

(2) *Monoclinic Sulphur*

Melt some sulphur in a small porcelain basin and allow it to cool slowly. As soon as a crust is formed

on the surface, pierce it with the end of a file or a thin rod and pour out the molten sulphur. A mass of shining transparent needles will now be found projecting from the cavity. This is called "prismatic," or monoclinic sulphur (Fig. 56. B). On standing, the crystals become opaque and revert to the permanent, stable rhombic form.

(3) *Plastic Sulphur* :

Take some powdered sulphur in a test tube and heat it gently. At first the sulphur begins to melt to a straw-coloured mobile liquid, which, as the temperature rises, grows dark and viscous, so much so that it will scarcely run out of the test tube even if it be held upside down. On prolonged heating, it again becomes somewhat mobile and finally it boils, giving off red vapours, which readily condense on a cool surface to minute yellow crystals. Now slowly pour the molten viscid sulphur in a thin continuous stream into a beaker of cold water. Remove the condensed sulphur from the water and examine it. Notice that it is no longer hard and crystalline but has been changed into a semi-transparent, amber-coloured, soft, stringy mass, which can be drawn out like caoutchouc. This is known as "plastic" sulphur. On keeping, it gradually reverts to the brittle yellow crystalline form which is more stable.

(4) *Milk of Sulphur* :

Boil a mixture of flowers of sulphur and slaked lime with water in a basin. When solution has taken place, filter the liquid. Add excess of hydrochloric acid to

the filtrate, and instantly the liquid becomes milky due to the separation of sulphur. This is known as "milk of sulphur." Collect a little of this in a filter paper, wash and dry. Shake up with carbon bisulphide, it is insoluble. Examine with a lens ; it is amorphous.

SULPHUR DIOXIDE

(1) Ignite a bit of sulphur in a deflagrating spoon and introduce it into a gas jar. It continues to burn for

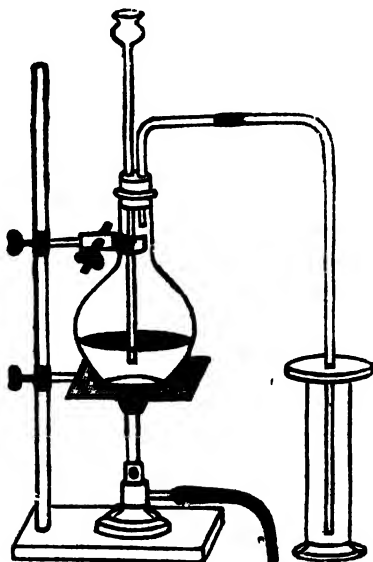


Fig. 57.

some time and then goes out. The jar no longer contains air but is filled up with a pungent smelling gas called sulphur dioxide. Add a little blue litmus solution, it is turned red.

(2) Fit up a flask with a thistle funnel and delivery tube as shown in Fig. 57. The lower end of the thistle funnel should reach very near to the bottom of the flask. Place some copper turnings in the flask and add strong sulphuric acid just to cover them. Apply gentle heat and collect the gas, which is evolved by downward displacement. Regulate the flame when the action is vigorous, so that the contents in the flask may not froth over.

A. Have three jars of the gas ready for testing the following properties.

(1) Introduce a lighted taper into one of the jars. It is extinguished.

(2) Invert a jar of the gas under water and take off the disc. Water slowly rises; the gas is readily soluble in water.

(3) Drop a red flower into a jar. The colour disappears, showing that sulphur dioxide has bleaching properties.

B. Pass the gas into some water in a test tube; it will dissolve. The solution smells of the gas and reddens blue litmus. To a little of the solution add some sodium carbonate; carbon dioxide is evolved, thus proving that sulphur dioxide forms an acid with water called sulphurous acid. To another portion of the solution add a little barium chloride solution—a white precipitate of barium sulphite is at once formed, which dissolves on the addition of a few drops of dilute hydrochloric acid.

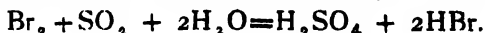
C. Allow the gas to bubble through the following solutions in separate test tubes and notice the effect in each case.

(1) Magenta solution is decolourised.

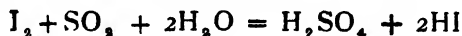
(2) A solution of potassium permanganate (KMnO_4) loses colour—the substance being reduced.

(3) A solution of potassium bichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) is reduced yielding a green solution.

4. Bromine water becomes colourless. Thus,



5. Iodine solution is decolourised. Thus,



D. Connect the delivery tube of your apparatus with a hard glass tube containing lead peroxide (PbO_2). On heating the oxide, and passing sulphur dioxide through the tube, the brown oxide is converted into a white substance, lead sulphate, (PbSO_4).

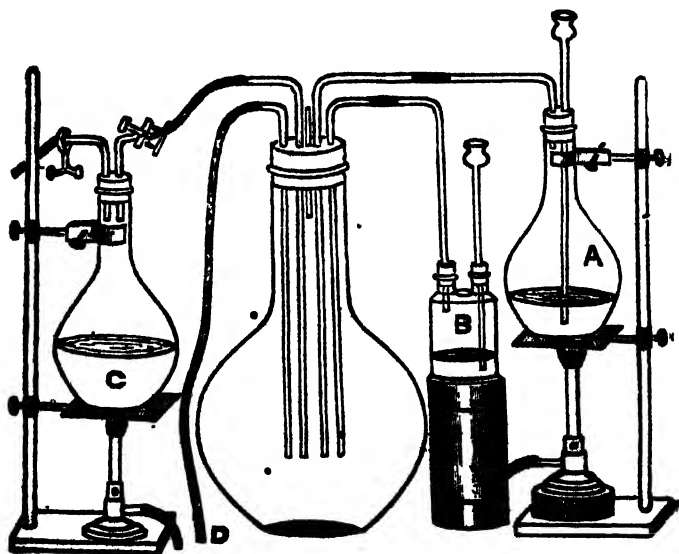
RECOVERY OF COPPER SULPHATE

After applying the above tests, detach the apparatus and pour out the contents of the flask into a beaker. Add some water and boil. Filter and evaporate the filtrate in a porcelain basin till a crust is seen to form on the surface of the liquid. Remove the flame and add water cautiously to redissolve the crust. Leave the solution in a crystallising dish in your cupboard till the next day, when blue crystals of copper sulphate will appear at the bottom of the dish. Remove the crystals

from the liquid and redissolve them in water and crystallise again. This time the substance will be purer than before. Remove the crystals from the mother liquor and press them between folds of blotting paper. Keep these corked up in a sample tube for future use.

SULPHURIC ACID

Fit up a large flask of about two litres capacity with a cork bored with five holes. Through these pass four leading tubes reaching near the bottom of the flask and



• Fig. 58.

• another short exit tube (Fig. 58). Through the leading tubes pass the following gases into the

(i) Sulphur dioxide from the flask A, generated from copper and sulphuric acid.

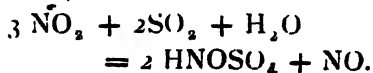
(ii) Nitric oxide from the bottle B, generated from copper and nitric acid.

(iii) Steam from the flask C. This flask is fitted up with two tubes closed by two screw cocks as shown in the figure, so that it is possible to cut off the steam supply by closing the tube on the side of the large flask and opening the other when steam will escape by this tube.

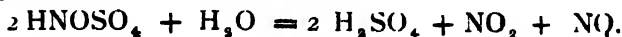
(iv) Air from a pair of foot bellows, by the tube D.

At the beginning, admit very little steam and you will notice that the sides of the flask are covered with a crystalline deposit of *chamber crystals*.

Thus,



These disappear as you admit more steam, generating sulphuric acid and nitrous fumes.



Continue the experiment till you have got a few c.c. of the acid collected at the bottom of the flask.

Now detach the apparatus and pour out the liquid in a small basin and heat with a small quantity of ammonium sulphate in order to drive off the dissolved nitrous gases. Allow to cool and apply the following tests.

(1) Take a few drops of the liquid in a test tube and dilute with water. Test with blue litmus; it is turned red. Add sodium carbonate; carbon dioxide is evolved.

(2) Heat another portion of the liquid with a few bits of copper turnings; sulphur dioxide is evolved.

(3) To a portion of the diluted acid add a little barium chloride solution. A white precipitate of barium sulphate is formed, which is insoluble in hydrochloric acid and even in aqua regia. This test distinguishes sulphates from sulphites.

SULPHURETTED HYDROGEN

You have noticed before that when iron sulphide is treated with an acid, a gas is evolved having the disagreeable odour of rotten eggs and the property of blackening a lead acetate paper. The gas can be prepared as follows.

Fit up an apparatus as in Fig. 42. Place some lumps of iron sulphide in the Woulff's bottle and pour dilute sulphuric acid down the thistle funnel. Action at once begins with effervescence. (A Kipp's apparatus may be more conveniently used). Collect several jars of the gas by downward displacement and apply the following tests.

(i) Pour a little blue litmus solution into a jar of the gas and shake well. The liquid is turned red, showing that sulphuretted hydrogen has acid properties.

(2) Pass a stream of the gas through cold water in a test tube. The water smells strongly of the gas. Add

a little lead acetate solution; a black precipitate is thrown down, showing that the gas is soluble in water.

(3) Have a jar of chlorine and bring another jar of sulphuretted hydrogen mouth to mouth with the first one and remove the discs. Fumes of hydrochloric acid are formed and a copious deposit of sulphur takes place on the sides of the jar



(4) Replace the delivery tube by a straight calcium chloride tube ending in a jet as in Fig. 59. Apply a light to the gas. It burns with a pale blue flame

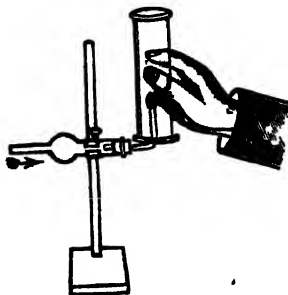
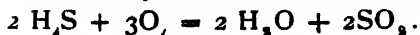


Fig. 59.

producing sulphur dioxide and water. The former is recognised by its smell and the latter is shown by bringing an inverted jar over the flame when the interior of the jar is covered with moisture.



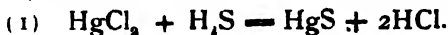
(5) Apply a lighted taper to a jar of the gas. The gas burns but the taper is extinguished and sulphur is deposited on the sides of the jar.



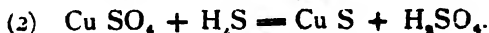
Now pour out the contents of the Woulff's bottle in a beaker and filter. Evaporate the filtrate in a basin and allow to crystallise. Green crystals of ferrous sulphate, $\text{Fe SO}_4 \cdot 7\text{H}_2\text{O}$, will separate out. Take these out of the mother liquor and press between folds of blotting paper.

PRINCIPLE OF QUALITATIVE ANALYSIS

Have in separate test tubes solutions of the following salts; (1) mercuric chloride, (2) copper sulphate, (3) antimony chloride, (4) zinc sulphate, (5) ferric chloride, (6) calcium chloride and (7) magnesium chloride. Add a few drops of dilute hydrochloric acid to each and pass a current of sulphuretted hydrogen from a Kipp's apparatus (see Fig. 37). Note that coloured precipitates are formed in some of them.



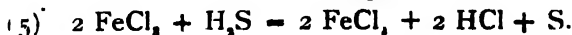
(Black)



(Black)

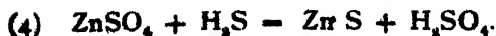


(Orange)

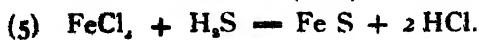


(deposit of "milk of sulphur").

In the case of ferric chloride, a precipitate of sulphur is thrown down, but if ferrous sulphate is used no precipitate will be formed. Similarly, no precipitate will be obtained in 4, 6 and 7. Now add enough ammonia, in each and at once precipitates are thrown down in 4 and 5, but 6 and 7 remain clear.



(White)



(Black)

The action of the ammonia is to neutralise the acids formed in the reactions. Thus we can divide the metals into three groups.

I. Those which give precipitates with sulphuretted hydrogen in acid solutions, *e.g.*, salts of mercury, copper, lead, bismuth, cadmium, arsenic, antimony and tin.

II. Those which give precipitates in alkaline solutions, *e.g.*, salts of aluminium, iron, chromium, zinc, manganese, nickel and cobalt

III. Those which remain unchanged in either case, *e.g.*, salts of calcium, barium, strontium, magnesium, potassium and sodium.

CHAPTER XV

OXIDATION AND REDUCTION

OXIDATION

Take a clean, dry porcelain crucible with a lid and support it on a clay pipe triangle placed on a tripod. Heat the crucible with the lid on by means of a Bunsen flame for about ten minutes. Then leave it to cool in a desiccator and weigh.

Put into the crucible a few bits of magnesium ribbon and weigh.

Heat the crucible with the lid on over a small Bunsen flame and when the magnesium begins to glow take away the flame. As soon as the glow has subsided, again apply heat. Now slowly turn the flame full and remove the lid from time to time to allow fresh air to enter and complete the oxidation. A portion of the oxide of magnesium is apt to be lost in the shape of fine dust ; hence particular care should be taken that it may not escape.

After heating for a quarter of an hour place the crucible in a desiccator to cool and weigh.

The operations of heating and weighing are repeated until the weight is constant. The results of an actual experiment are entered as follows :—

Wt. of crucible with lid,	...	= 16.42 grams.
Do. + magnesium	...	= 16.65 „

∴ Wt. of magnesium ... = 0.23 „

Wt. of crucible with lid + oxide = 16.80 „

∴ Wt. of oxide ... = 0.38 „

∴ Increase in weight of magnesium = 0.15 „

This represents the amount of oxygen taken up by the magnesium.

Calculate the percentage of magnesium in the oxide, thus :

0.38 ∴ 100 :: 0.23 :	per cent of magnesium
Magnesium	... = 60.5
Oxygen	... = 39.5
	<hr/>
	100.0

Theoretically,

Magnesium	... = 60
Oxygen	... = 40
	<hr/>
	100

Repeat the above experiment with a few bits of tin foil and calculate as follows :—

Wt. of crucible with lid .. = ... grams .

Do + tin ... = ... „

Wt. of tin ... = ... „

Do crucible with lid + oxide = ... „

Wt. of crucible with lid ... = ... „

∴ Wt. of oxide ... = ... „

∴ Wt. of tin ... = ... „

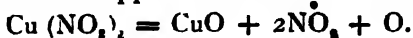
∴ Wt. of oxygen (by difference) = ... „

Whence calculate the percentage of tin in tin oxide.

Here you have seen that some metals increase in weight when calcined in air and that the process can be carried to completion simply by heating the substance in air. But many other metals are not so easily oxidised. Copper and lead, for instance, when heated in air, are not completely oxidised ; but a film of oxide covers the surface, which prevents further oxidation.

But metals, such as these can usually be completely oxidised by heating them with certain substances very rich in oxygen, a portion of which they can readily give up. These when employed for the purpose of oxidation are called *oxidising agents*. Those usually employed in the laboratory are oxygen, nitric acid, potassium chlorate, potassium nitrate, etc.

Weigh out about 2 grams of copper turnings in a small basin and add a little nitric acid just sufficient to dissolve the copper, forming a blue solution of copper nitrate. Transfer it to a crucible, which has been previously weighed with the lid. Rinse the basin with water into the crucible and evaporate to dryness on a water bath. Place the crucible with the lid on over a triangle and at first very gently heat it by means of a naked flame. Unless this precaution is taken, particles of the dry copper nitrate will be lost by spitting. After a few minutes heat more strongly. Note that reddish brown nitrous fumes are given off and a black residue of copper oxide is left in the crucible. Thus,



Now cool the crucible in a desiccator and note the weight. Heat once more, cool as before and weigh it again. The two weights must be constant. Enter your results as follows :

Wt. of copper taken	=	... grams.
Wt. of crucible with lid	=	... „
„ + CuO	=	... „
∴ Wt. of CuO	=	... grams.
Wt. of copper	=	... „
Wt. of oxygen (by difference)	=	... grams.

Whence calculate the percentage of copper in copper oxide.

There are several instances where an apparent diminution in weight takes place by calcining the substances in air or oxygen.

Take a clean, dry, porcelain crucible, as before, and weigh. Put a little charcoal powder in it and again weigh. Heat the whole by a Bunsen flame. After heating for some time, when all the charcoal has been burnt to ashes, cool it in the desiccator and weigh. You will notice a loss of weight which is due to the carbon having escaped as carbon dioxide.

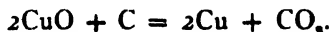
If you can absorb the products of oxidation, *viz.*, carbon dioxide, by a suitable absorbent, you will notice an increase in weight, owing to the combination of carbon (charcoal) with the oxygen of the air. (Cf. Exp. 12, Inorganic Chemistry, pp. 12-13).

REDUCTION

Take a little of the dry black substance, copper oxide, prepared before, in a small mortar and mix it thoroughly with about an equal amount of charcoal powder.

Heat a portion of the mixture in a hard glass test tube fitted with a cork and a delivery tube, which dips into a test tube containing clear lime water, as shown in Fig. 52.

By and by you will see that the whole mass has become red due to the formation of particles of metallic copper and the lime water has turned milky, showing that carbon dioxide has also been evolved in the reaction.



This process of obtaining a metal from its oxide is called *reduction*, and substances like charcoal, employed in this experiment, which can take up the oxygen from an oxide, *i. e.*, deoxidise it, are called *reducing agents*. Hydrogen, carbon monoxide and coal gas are some of the reducing agents used in the laboratory.

CHAPTER XVI

EQUIVALENTS

DETERMINATION OF THE EQUIVALENT OF ZINC

Take about 0.1 gram of chemically pure zinc accurately weighed out in a balance and put it on a watch glass which should be placed in a beaker. Cover the bit of zinc with a small funnel. Fill the beaker with

water which will rise to a height of about two inches above the stem of the funnel. Take a long graduated tube sealed at one end, measuring up to 100 c.c. Fill the tube completely with water, close the open end with the thumb and lower it into the water in the beaker so as to make the tube surround the stem of the funnel. Clamp it in a vertical position (Fig. 60). Now allow a few c. c. of strong sulphuric acid to come in contact with the zinc by means of a pipette. At first no action is observed ,

but on adding by means of another pipette a few drops of platinum chloride or copper sulphate solution the evolution of a steady stream of gas is observed, which collects in the upper part of the graduated tube.

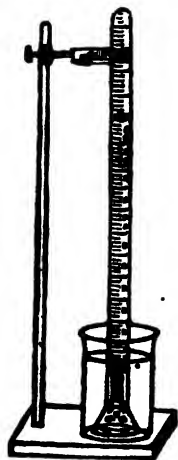


Fig. 60.

Wait till all the zinc is dissolved. If the action ceases before the whole of the zinc is used up add a little more of the acid when the action will commence again. After the collection of all the hydrogen, close the open end with your thumb and take out the tube. Plunge it in a deep trough or tall gas jar full of water. Make the level of water inside the tube same as the level outside, and carefully read off the volume of the gas (Fig. 61). Never hold the tube directly with your fingers but use a small clamp or a strip of paper wrapped round the tube as the warmth of the hand will change the volume of the gas. Determine the temperature of the water by a thermometer and the atmospheric pressure by a barometer. Having thus obtained

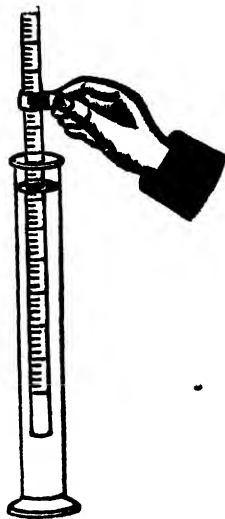


Fig. 61.

the volume, temperature and pressure of the moist gas, calculate the volume of the gas at N.T.P. from the formula given below;

$$\frac{V}{V'} = \frac{273}{273+t} \times \frac{p-x}{760}$$

where V is the volume of the gas at N.T.P. and V' the observed volume at temperature $t^{\circ}\text{C.}$ and p the barometric pressure and x the aqueous tension at $t^{\circ}\text{C.}$ Thus

$$V = V' \times \frac{273}{273 + t} \times \frac{p - x}{760}$$

Now the weight of this volume of hydrogen at N.T.P. can be easily calculated as the weight of a litre of this gas at N.T.P. is 0.09 gram.

From the above results the equivalent of zinc is calculated by dividing the weight of the zinc taken by the weight of the liberated hydrogen.

DETERMINATION OF THE EQUIVALENT OF MAGNESIUM

Take a piece of pure bright magnesium ribbon, *i.e.*, free from all oxide and weigh out about 0.05 grams. Place the coiled ribbon on a watch glass and proceed exactly as described under zinc. On the addition of a few c. c. of sulphuric acid, the magnesium begins to dissolve evolving hydrogen, which is collected in the graduated tube. When the whole of the magnesium has been dissolved, the volume which the liberated hydrogen occupies at N.T.P. is calculated and its weight determined as before. Dividing the weight of the magnesium by the weight of the hydrogen, the equivalent of the metal is determined.

The equivalent of magnesium may also be determined by converting a weighed quantity of it into oxide in a covered crucible and determining the weight of the resulting oxide. (Vide Chapter XV, pp. 99-100). The amount of magnesium which will combine with 8 parts by weight of oxygen will be the equivalent of the metal

as you know that 8 parts by weight of oxygen combine with 1 part by weight of hydrogen.

DETERMINATION OF THE EQUIVALENT OF COPPER

The equivalent of copper may be determined by first converting a known weight of pure copper gauze into nitrate and then into oxide using proper precautions as directed in p.p. 101-102, or by reducing a known weight of dry copper oxide in a current of dry hydrogen or coal gas which contains both carbon and hydrogen, which are good reducing agents.

Take some black copper oxide in a dry porcelain crucible, place it on a clay pipe triangle over a tripod

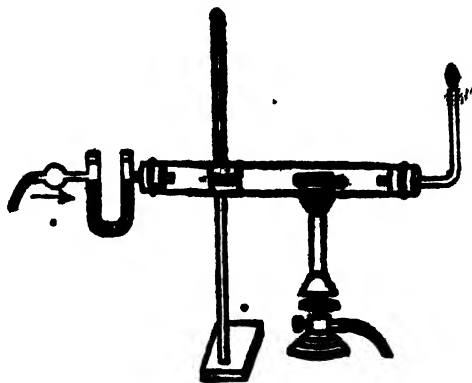


Fig. 62.

and heat it for some minutes in order to expel all moisture, as copper oxide is highly hygroscopic. Cool the crucible in a desiccator and weigh out about 1 gram of

copper oxide in a small porcelain boat of known weight. Introduce this into a combustion tube about a foot long corked at both ends through which hydrogen from a Kipp's apparatus or coal gas from the gas tap can be passed as shown in Fig. 62. If hydrogen be used, it should be purified and dried by successively passing it through two wash bottles containing potassium permanganate solution and strong sulphuric acid. In the case of coal gas, it should be passed through a U tube containing crystals of lead acetate in one limb and fused calcium chloride in the other. The limb containing lead acetate is connected with the gas supply. Thus traces of sulphuretted hydrogen in the gas are absorbed by lead acetate and moisture by calcium chloride. Clamp the tube in an inclined position as shown in the figure and pass a current of coal gas through it. Ignite the gas at the drawn out end of the tube when free from air and then begin to heat the copper oxide strongly in a small gas furnace or by means of a Teclu burner. After some time the black copper oxide will be reduced to red metallic copper powder. Allow the tube to cool in a current of the gas. When it is cold, take out the boat and weigh. Then heat it again in a current of the gas, cool and weigh as before. Repeat the operations till no further loss of weight is noticed. Enter the results thus :

1)	Weight of the boat	...	—	...	grms.
(2)	Do + Cu O	...	—	...	"
(3)	Wt. of Cu O	...	—	...	"

- (4) Wt. of boat + reduced copper = ... ,
(5) Wt. of reduced copper ... = ... ,,
Wt. of oxygen which has,
combined with copper = (3) - (5) = ... ,,

The equivalent of copper is obtained by calculating the weight of copper which will combine with 8 parts by weight of oxygen.

CHAPTER XVII

ACIDIMETRY AND ALKALIMETRY VOLUMETRIC ANALYSIS

You know that alkalies and acids neutralise each other in certain proportions forming salts. This principle has been utilised in measuring the amount of acid or alkali present in a given solution. The plan adopted is this:—a solution of an alkali or acid of known strength is first prepared, and then the unknown acid is measured by the known alkali (*acidimetry*) or the unknown alkali measured by the known acid (*alkalimetry*). In these processes the solutions are universally measured by volumes and hence this branch of chemistry is known as *volumetric analysis*.

These solutions of known strength are also called *standard solutions*. These might be prepared of any known strength. But, for the sake of convenience they are prepared in such a way that equal volumes of an acid and an alkali will neutralise each other. For this purpose, the substances are taken in proportion to their equivalents. A solution which contains one equivalent in grams per litre is called a *normal solution*. Thus, the molecular weight of NaOH is 40 and it contains one equivalent of Na , *i.e.*, 23 grams; and hence 40 grams of NaOH contained in a litre of the solution would make a normal solution of caustic soda.

A solution one tenth 'as strong or in other words containing as much of the substance as one tenth of that used in preparing a normal solution, is called a *decinormal* or N/10 solution. For ordinary estimations these solutions are used in preference to the normal solutions. Solutions as weak as N/100 are sometimes used for special purposes.

DECINORMAL SODIUM CARBONATE *i.e. a decinormal alkaline sol*

The molecular weight of Na_2CO_3 is 106 and one molecule of it contains 2 equivalents of sodium. Hence *(i.e. 106)* 53 grams of it would be required to make a litre of normal solution, and 5.3 grams for decinormal solution.

Take some pure anhydrous sodium carbonate in a basin and heat for a few minutes by a small Bunsen flame stirring with a glass rod. Take care that the substance does not fuse. Then cool in a desiccator and weigh out accurately 5.3 grams of the dry substance in another basin. Have a clean litre measuring flask. Dissolve the substance in a little distilled water and transfer the solution into the measuring flask. Wash the basin several times with distilled water and add the washings to the flask. When the last trace of sodium carbonate has been transferred to the flask, add water up to the mark. Thus you get one litre of N/10 solution of Na_2CO_3 .

DECINORMAL SULPHURIC ACID *i.e. a decinormal acid solution*

The molecular weight of H_2SO_4 is 98 and one molecule of it contains 2 equivalents of hydrogen. Hence,

4.9 grams of it would be required for a litre of decinormal solution. In order to prepare this first take the specific gravity of the acid and find out the strength from a table and calculate the volume which is to be taken for a solution. For example, the specific gravity of the pure strong acid is say 1.84, and from the table you see that it contains 95.6 grams of H_2SO_4 per 100 grams of the liquid. To calculate the volume proceed thus :

100 grams of the acid occupy $100 \div 1.84 = 54.34$ c.c.
and this volume contains 95.6 grams of H_2SO_4 .

$$95.6 : 4.9 :: 54.34 \cdot x \text{ c.c.}$$

$$x = \frac{54.34 \times 4.9}{95.6} = 2.8 \text{ c.c. nearly}$$

Hence take out the acid in a fractional pipette which is a glass tube drawn out at one end, of 10 or 5 c.c. capacity, each c.c. being divided, into one tenth along the entire length of the tube, and deliver 2.8 c.c. of the acid in a litre measuring flask. Now add water up to the mark. Thus you get an approximate N/10 solution of H_2SO_4 . In order to ascertain the accurate strength of this acid, fill a burette with it see directions in Chapter II) and by means of a pipette transfer 10 c.c. of N/10 Na_2CO_3 solution in a clean porcelain basin. Add two or three drops of methyl orange solution*; the solution will be turned yellow. Now go on adding the acid drop by drop from the burette and stir

* Methyl orange imparts a slight yellow colour to liquids containing excess of alkali which turn pink by a slight excess of acid.

with a glass rod until you just get a pink colour. This operation is called *titration*. Read off the burette and note it down. Again fill the burette to the zero mark and repeat the titration three or four times using 10 c.c. of the alkali each time and take the mean of the readings. The several readings should not differ by more than 0.1 c.c.

Calculations :

Suppose 9.8 c.c. of the acid is required to neutralise 10 c.c. of the alkali. Therefore, 980 c.c. of this is equivalent to 1000 c.c. of N/10 Na_2CO_3 . Hence, 980 c.c. of this acid must be diluted to 1000 c.c. by adding more water in order to make it accurately decinormal. Or, you may find out the factor thus:

$$980 \text{ c.c.} = 1000 \text{ c.c. of N/10 } \text{Na}_2\text{CO}_3 \text{ solution.}$$

$$\text{Hence, } 1 \text{ c.c.} = 1.0204.$$

DECINORMAL CAUSTIC SODA 100 cc. N/10 NaOH

Weigh out about 4 grams of pure caustic soda in a watch glass and dissolve in water previously boiled and make up to 1000 c.c. in a measuring flask. Transfer 10 c.c. of this solution to a basin, add a few drops of litmus solution and titrate with N/10 H_2SO_4 and find out the strength.

Calculations :

Suppose 9.5 c.c. of N/10 H_2SO_4 , whose factor is 1.0204, is required to neutralise 10 c.c. of the alkali; and 9.5 c.c. of the above acid is equivalent to

$$9.5 \times 1.0204 = 9.7 \text{ c.c. N/10 solution. Hence, 9.7 c.c. is}$$

equivalent to 10 c.c. of the alkali and the factor is $9.7 \div 10 = 0.97$.

DECINORMAL HYDROCHLORIC ACID *(c. decinormal acid)*

The molecular weight of hydrochloric acid is 36.5. A molecule of this acid contains one equivalent of hydrogen. Hence, 3.65 grams of it will be required to make a N/10 solution. Strong hydrochloric acid is an aqueous solution of the gas. Hence, in order to prepare the solution, first take the specific gravity of the strong acid and from the table (Appendix IV) find out its strength and calculate the volume which would contain 3.65 grams of HCl gas. Now deliver the calculated amount of the strong acid into a measuring flask by means of a fractional pipette and make up to 1000 c.c. with distilled water. Titrate with decinormal caustic soda solution using litmus as an indicator, and find out the factor.

PROBLEMS

1 To determine the strength of a sample of sulphuric acid. *

Take out 5 c.c. of the given acid and dilute up to 250 c.c. in a measuring flask of that size.

Fill your burette with this acid and titrate with N/10 NaOH, using litmus as indicator. Take the mean of three titrations.

Suppose 10.5 c.c. of this acid is required to neutralise 10 c.c. of N/10 alkali (factor—0.97).

* The teacher should give the student a sample of acid to determine its strength.

10 c.c. of the alkali = 9.7 c.c. of N/10 alkali.

10.5 c.c. of the acid is equivalent to 9.7 c.c. of N/10 alkali.

$$\therefore 1 \text{ c.c.} = \frac{9.7}{10.5}, \text{ or } 250 \text{ c.c.} = \frac{9.7 \times 250}{10.5}.$$

Now, 1 c.c. of N/10 H_2SO_4 contains $\frac{4.9}{1000}$ or 0.0049 grams of H_2SO_4 . Hence, in 250 c.c. of the acid we have $\frac{9.7 \times 250}{10.5} \times 0.0049$ grams of H_2SO_4 .

This is contained in 5 c.c. of the original sample. Therefore, the above number must be multiplied by 20 to calculate the amount of H_2SO_4 in grams per 100 c.c. of the sample.

2. To determine the amount of total alkali in soda ash (calculated as Na_2CO_3).

Weigh out about 2 grams of the sample of the substance from a weighing bottle and dissolve in water and make the solution up to 250 c.c. in a measuring flask. If there be any insoluble impurities, allow them to settle. •

Take out 10 c.c. of the clear solution by means of a pipette in a basin. Dilute with water. Add a drop of methyl orange. Titrate with N/10 HCl which is added from a burette. • Take the mean of three or four titrations as before. From the amount of acid required, calculate the amount of Na_2CO_3 in the substance.

• Suppose 12.5 c.c. of the solution of the substance is required to neutralise 10 c.c. of N/10 HCl.

Then 12.5 c.c. = 10 c.c. N/10 HCl.

$$\text{Or, } 1 \text{ c. c.} = \frac{10}{12.5} \text{ c.c. N/10 HCl}$$

$$\therefore 250 \text{ c.c} = \frac{10 \times 250}{12.5} \text{ c.c. N/10 HCl.}$$

Now 1 c.c. of N/10 Na_2CO_3 solution contains 0.0053 grams of Na_2CO_3 . Hence, in 250 c. c. of the solution we have

$$\frac{10 \times 250}{12.5} \times 0.0053 \text{ grams of } \text{Na}_2\text{CO}_3.$$

This is contained in 2 grams of the original sample, and hence calculate the amount in 100 grams.

CHAPTER XVIII

COMBUSTION—STRUCTURE OF FLAME—BLOWPIPE

COMBUSTION

When a magnesium ribbon is heated, in the air, it takes fire and burns with dazzling brilliancy. Considerable evolution of heat and light takes place and a white residue is left behind.

Again, take a bit of sulphur in a crucible and heat it, and when it takes fire, remove the flame. The sulphur continues to burn with a bluish flame and at the same time a suffocating smell is noticed. In a few minutes all the sulphur is burnt away as sulphur dioxide and nothing is left behind.

These are instances of combustion by which we generally mean the combination of substances with oxygen with evolution of heat and light.

But in its widest application, the term is applied to any chemical change attended with heat and light, *e. g.*, hydrogen burning in chlorine.

SLOW COMBUSTION

The phenomenon of the rusting of iron filings is an example of slow combustion. The union of iron with the oxygen of air takes place very slowly and the heat which is evolved is lost by radiation or conduction as soon as it is produced and there is no appreciable rise

in temperature. But when iron burns in oxygen the union takes place very rapidly and the iron becomes incandescent.

When a piece of lime is held in an oxyhydrogen flame or a platinum wire is heated, the temperature gradually rises until at last the substance becomes incandescent. This phenomenon is not combustion because although the substance emits heat and light, it does not undergo any chemical change. It regains its original appearance, as soon as it is removed from the source of heat. *

EXPLOSION

This is an instance of combustion, in which so much heat is evolved in a moment, as to increase enormously the pressure of the gases produced by the action, giving the surrounding atmosphere a sudden shock causing a loud report.

A mixture of two volumes of hydrogen and one of oxygen collected in a detonating bottle and brought near a flame explodes, accompanied by a sudden flash with a very loud report.

COMBUSTIBLE SUBSTANCES AND SUPPORTERS OF COMBUSTION

In the ordinary cases of combustion, *e. g.*, coal gas or hydrogen burning in air, we call the substances

* Oxides of aluminium, magnesium, barium and strontium are examples of substances, which become incandescent, like lime, when heated in air, without undergoing any chemical change.

burning as the combustible substances and the surrounding envelope of air as the supporter of combustion. But these terms are relative and the process may be reversed by a slight modification of the conditions, as will be proved by the following experiments.

(1) Take an ordinary lamp chimney and fit its lower opening with a cork bored with two holes. Through one hole pass a short glass tube, about 7 or 8 m.m. diameter and about 10 c.m. long. Through the other pass another glass tube, about 4 or 5 m.m. diameter and connect it with a gas tap by means of a rubber tubing (see Fig. 63). Clamp the arrangement upright and cover the top of the chimney with a mica plate with a hole in the centre. Turn the gas tap. As the stream of gas passes through the narrow tube, air will be drawn in through the wider one. Now pass a lighted taper up the wider tube when the air will take fire and burn in the atmosphere of coal gas with a pale blue flame. Here, the air may be called the combustible substance and coal gas the supporter of combustion.



Fig. 63.

The excess of coal gas escaping from the top can be inflamed when it burns with a luminous flame. In this case, the coal gas is the combustible substance and air the supporter of combustion. So these terms are relative.

(2) Connect a glass tube ending in a jet to a gas holder containing oxygen. On slowly opening the tap the oxygen escapes through the jet in a narrow stream. Now take a tall jar full of hydrogen and hold it mouth downwards. Ignite the gas at the mouth and immediately thrust the jet in. (Fig. 64). The jet of oxygen ignites as it passes the zone of burning hydrogen and continues to burn until all the hydrogen is used up.

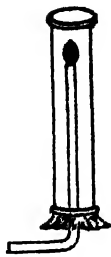


Fig. 64

STRUCTURE OF FLAME

The Bunsen burner, besides being used for ordinary heating purposes, constitutes a most valuable analytical instrument. Its special properties depend upon the fact that different regions of the flame possess different temperatures and it may be adapted for oxidation as well as for reduction. So by a judicious use it may be made to serve various purposes.

In order to facilitate the application of the various reactions, a brief outline of the structure of the Bunsen flame is given here. The Bunsen flame when it is getting a full supply of air consists of two cones. The inner cone is darker in colour and consists of hot unburnt gas. The outer hollow cone is light-blue in colour and it is here that the gas burns. That the inner cone consists of unburnt gas may be easily proved by various experiments.

(1) Pierce a match stick with a pin. Place the pin across the mouth of the burner so that the head of the match stick projects a little above the mouth of the burner (Fig. 65). Light the gas; it burns as usual but the match stick does not take fire.

(2) Take a sheet of white paper and depress it upon Bunsen flame momentarily. The paper will be charred in a circle leaving the central portion quite clean.



Fig. 65.



Fig. 66.

Partially close the air holes. The tip of the inner cone will appear luminous. This luminous region has got reducing properties while the non-luminous outer cone has got oxidising properties (Fig. 66).

Now as it is not always convenient to hold the substance directly in the flame, for oxidising or reducing purposes, the flame is directed against the substance conveniently placed by means of a mouth blowpipe.

For this purpose a jet is placed in the burner and the air holes are closed. After lighting the gas, the mouth of the blowpipe is placed on the tip of the jet and air blown in slowly and uniformly. Here the student should practice how to blow a continual and steady current of air through the pipe. The air should not be blown from the lungs but from the cheeks inflated with air. A slight practice will render the process easy.

Now the flame shall assume the shape as shown in Fig. 67. When it is required to oxidise something it is



Fig. 67.

held at *o*, the tip of the outer cone. While, on the other hand, if it is required to be reduced, the substance is held at *r* near the tip of the inner cone. The different effects may be seen from the following experiment.

Have a piece of glass known as lead glass, so named because it contains some lead salt. Hold a point of the glass against the reducing flame. It will presently assume a shining black appearance owing to the reduction of the lead salt to metallic lead. Now hold the

blackened spot against the oxidising flame, and it will again become colourless.

BLOWPIPE REACTIONS

After having acquired some practice in the use of the mouth blowpipe, the student should proceed with the following dry reactions

LEAD

(a) *Reduction* : Mix some lead oxide or litharge with dry sodium carbonate in a mortar. Place a small quantity of this mixture in a shallow depression made on a piece of sound charcoal by means of a penknife or cork borer. Close the air holes of your Bunsen burner, insert the blowpipe jet within the tube and light the gas. Direct a gentle blast of air across the flame from the nozzle of your blowpipe, and hold the substance in the reducing flame. In a short time, the substance will melt, and if the blowing is continuous, small metallic globules or beads of lead will be observed. The beads, on being hammered on an anvil, are found to be malleable and may be beaten into thin foils. Notice also that metallic lead is very soft and leaves a black mark on paper.

(b) *Oxidation* : Heat the small metallic beads of lead again on a piece of charcoal in the oxidising flame. Observe the yellow oxide of lead formed as a crust and the shining metallic lustre is no longer visible.

COPPER

Mix a small quantity of black copper oxide or any other copper compound with excess of dry sodium carbonate and potassium cyanide, and heat the mixture on a piece of charcoal in the reducing flame. After continuous blowing for some minutes small reddish particles or scales are observed. Dissolve these particles in nitric acid and notice the blue solution of copper nitrate, which becomes intensely blue on the addition of excess of ammonium hydrate.

MERCURY

Mix a little mercuric chloride or any other mercury compound with dry sodium carbonate and heat the mixture on a piece of charcoal in the reducing flame. Observe that the substance volatilises and no metallic beads are formed. Heat the same mixture in a bulb tube in the Bunsen flame and notice that metallic mercury sublimes and forms a mirror in the cold part of the tube. The bulb tube may be broken and the minute globules of mercury examined on a piece of paper or watch glass.

IRON

Mix some ferric oxide or any other iron compound with sodium carbonate and heat the mixture on a piece of charcoal in the reducing flame. Observe that a black residue is left behind, which is attracted by a magnet. Treat the powder with strong HCl and a few drops of HNO_3 , warm and add to the yellow solution a few drops

of potassium ferrocyanide solution and notice the fine blue precipitate known as Prussian blue.

ZINC

(a) Heat a small quantity of a mixture of sodium carbonate and zinc carbonate or any other compound of zinc on a piece of charcoal in the reducing flame. Observe that no metallic beads are obtained, as the metal volatilises and leaves behind an incrustation of zinc oxide, which is yellow when hot and white when cold.

(b) Heat some zinc oxide or any other compound on a piece of charcoal in the oxidising flame (without the addition of any reducing agent) moisten the residue with a few drops of cobalt nitrate solution and heat again in the oxidising flame. Observe that a beautiful green mass is left behind.

MAGNESIUM

Heat some magnesium carbonate on a piece of charcoal in the oxidising flame after moistening with cobalt nitrate solution and observe that an infusible pink residue is left behind.

BORAX BEAD TESTS

Take a platinum wire with one end fused to a glass rod and the other end bent in the form of a loop. Heat the wire in the oxidising flame and while red hot dip it in some powdered borax in a watch glass. Some borax will adhere to the loop. Heat the borax again

and observe that it swells up and finally becomes colourless and transparent. Fuse this transparent borax bead with a trace of copper oxide in the oxidising flame; the bead will be greenish blue, which, when heated again in the reducing flame, becomes almost colourless.

Prepare another colourless borax bead, after throwing away the copper bead and cleaning the wire, and fuse it with a trace of ferric oxide in the oxidising flame. The bead will be coloured light yellow when cold and when heated again in the reducing flame will be changed to light green (bottle green).

X FLAME COLOURATIONS (*Done... modified at 30th July 1914*)

Take a little of the substance in a watch glass, moisten with a few drops of hydrochloric acid. Heat a trace of the substance at the end of a platinum wire in the outer mantle of the Bunsen flame and note the colour of the flame.

Sodium Compounds—Intense yellow.

Calcium ,, — Red.

Copper , — Green.

1. *N. B.* The platinum wire should be properly cleaned after each experiment by repeatedly dipping it in hydrochloric acid and heating in the Bunsen flame.

2. *Other metals viz. Mg, Fe, Zn, Hg, give the colouration as in a non-luminous flame*

CHAPTER XIX

WET REACTIONS

MERCURY

It forms two series of salts, mercurous and mercuric.

Mercurous salts :

Use a solution of mercurous nitrate.

(1) HCl or soluble chlorides produce a white precipitate of mercurous chloride or calomel, Hg_2Cl_2 , which is insoluble in HNO_3 and is blackened by NH_4OH .

(2) H_2S produces a black precipitate of mercuric sulphide and metallic mercury. This black sulphide is insoluble in HNO_3 and soluble in aqua regia.

(3) NaOH or KOH produces a dark brown or black precipitate of mercurous oxide.

(4) SnCl_2 precipitates grey metallic mercury. If the liquid be poured off and the grey residue boiled with strong hydrochloric acid, distinct globules of mercury are obtained.

(5) KI produces a green precipitate of mercurous iodide, Hg_2I_2 .

(6) Place a bit of bright copper in a solution of mercurous or mercuric salt; notice that the copper is coated with a grey deposit of mercury, which becomes bright on rubbing.

Mercuric Salts :

Use a solution of mercuric chloride.

(1) H_2S produces a black precipitate of HgS , mercuric sulphide. If H_2S is passed very slowly or better when an aqueous solution of H_2S is added gradually to a dilute solution of the mercuric salt, at first a white precipitate is observed, which rapidly changes to yellow, brown, red and finally to black sulphide on the addition of excess of the reagent. These coloured precipitates are due to mixtures of HgS and $HgCl$ in varying proportions. HgS is insoluble in HNO_3 , but soluble in aqua regia

(2) $NaOH$ or KOH produces a yellow precipitate of mercuric oxide.

(3) NH_4OH produces what is known as "infusible white precipitate," $NH_4 HgCl$.

(4) KI gives a scarlet precipitate of HgI_2 , mercuric iodide, soluble in excess of either of the reagents.

(5) $SnCl_2$ produces at first a white precipitate of mercurous chloride, which is reduced to grey mercury on the addition of excess of the reagent. Boil the grey residue with strong HCl ; globules of mercury will be obtained.

COPPER

Use a solution of copper sulphate.

(1) H_2S precipitates black cupric sulphide, soluble in hot nitric acid and in potassium cyanide but insoluble in caustic alkalis and also in hot dilute H_2SO_4 .

(2) NaOH or KOH produces a greenish blue precipitate of cupric hydrate, which turns to black CuO on boiling with excess of the reagent. In the presence of grape sugar the blue precipitate of $\text{Cu}(\text{OH})_2$ on boiling, is gradually changed into red cuprous oxide Cu_2O .

(3) Dilute ammonia when added cautiously in small quantities produces at first a greenish blue precipitate of copper hydrate, which is soluble in excess of the reagent, forming a beautiful azure blue liquid.

(4) $\text{K}_4\text{Fe}(\text{CN})_6$, potassium ferrocyanide, produces even in very dilute solutions a reddish brown (chocolate) precipitate of cupric ferrocyanide.

IRON

It forms two series of salts, ferrous and ferric.

Ferrous Salts :

Take some green crystals of ferrous sulphate or green vitriol and dissolve in distilled water, previously boiled to expel all dissolved air. Any yellow crust of ferric sulphate should first be removed by repeated washing with distilled water. Use a solution of ferrous sulphate thus prepared.

(1) $(\text{NH}_4)_2\text{S}$, ammonium sulphide, produces a black precipitate of ferrous sulphide, readily soluble in acids with the evolution of H_2S .

(2) H_2S gives no precipitate in acid solutions; in dilute neutral solutions FeS is partly precipitated.

(3) Ammonia produces a white precipitate of ferrous hydrate, when the ferrous salt is free from ferric salt;

this precipitate turns rapidly to a dirty green colour owing to the absorption of atmospheric oxygen.

Ferric Salts :

Use a solution of ferric chloride.

(1) H_2S , in acid solution, produces a precipitate of sulphur, and the salt is reduced to ferrous salt.

(2) $(\text{NH}_4)_2\text{S}$ produces a black precipitate of FeS , ferrous sulphide; the ferric salt being reduced to ferrous salt with separation of sulphur.

(3) NH_4OH , or alkaline hydrate, produces a reddish brown precipitate of ferric hydrate readily soluble in acids.

DISTINGUISHING TESTS FOR FERROUS AND FERRIC SALTS

Reagents	Ferrous salts	Ferric salts
Potassium ferrocyanide solution. $\text{K}_4\text{Fe}(\text{CN})_6$	produces a bluish white precipitate of $\text{K}_2\text{F}_6(\text{CN})_6$ which is rapidly changed to Prussian blue on exposure to air.	a fine blue precipitate. (Prussian Blue) Insoluble in HCl but soluble in oxalic acid ; decomposed by NaOH with formation of ferric hydrate.
Potassium ferricyanide solution $\text{K}_3\text{Fe}(\text{CN})_6$	a dark blue precipitate (Turnbull's Blue)	no precipitate, but simply changes the colour to reddish brown
Ammonium or potassium sulphocyanide solution.	no colouration if the salt is perfectly free from ferric.	a blood red colouration even in very dilute solutions.

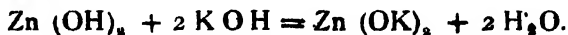
ZINC

Use a solution of zinc sulphate, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$

(1) Ammonium sulphide, $(\text{NH}_4)_2\text{S}$, gives a white precipitate of zinc sulphide, ZnS ; soluble in HCl and H_2SO_4 with the evolution of H_2S .

(2) H_2S produces in neutral solutions a partial precipitate of ZnS .

(3) NaOH or KOH precipitates the hydrate, $\text{Zn}(\text{OH})_2$; soluble in excess of the reagent forming a zincate.



CALCIUM

Use a solution of calcium chloride, CaCl_2 .

(1) Ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3$, produces a white precipitate of CaCO_3 .

(2) H_2SO_4 or a soluble sulphate produces, from a strong solution of calcium chloride, a white precipitate of calcium sulphate, CaSO_4 , sparingly soluble in water. It dissolves on boiling in a strong solution of ammonium sulphate.

(3) Ammonium oxalate, $\text{C}_2\text{O}_4(\text{NH}_4)_2$, produces, even from a very dilute solution, a white crystalline precipitate of calcium oxalate, soluble in mineral acids (HCl or HNO_3), insoluble in acetic acid, $\text{C}_2\text{H}_3\text{O}_2$.

MAGNESIUM

Use a solution of magnesium sulphate.

(1) NH_4OH produces a partial precipitate of magne-

sium hydrate $\text{Mg}(\text{OH})_2$ in the absence of ammonium chloride. It gives no precipitate in the presence of NH_4Cl owing to the formation of a soluble double chloride.

(2) Baryta water, $\text{Ba}(\text{OH})_2$, also precipitates $\text{Mg}(\text{OH})_2$ in the absence of ammonium salts.

(3) Sodium hydrogen phosphate, Na_2HPO_4 , produces a white crystalline precipitate of ammonium magnesium phosphate, MgNH_4PO_4 , in the presence of ammonium chloride and ammonia; this precipitate is soluble in dilute mineral acids and also in acetic acid. From very dilute solutions the precipitate is slowly obtained after rubbing the sides of the test tube with a rod.

SODIUM

All sodium compounds are soluble in water, and impart an intense golden yellow colour to the non-luminous Bunsen flame.

EXAMINATION OF SOLIDS IN NATURAL WATERS

(QUALITATIVE)

Take some water to be examined in a beaker and boil. When about three fourths of the liquid have been evaporated, add more of the water and boil again. In this way a large volume of the water will be boiled off and when finally evaporated to dryness in a porcelain basin on a water bath, will leave behind a fairly large quantity of solid residue. Dissolve this residue in distilled water, filter off any insoluble matter and test the clear filtrate

by the following tests. Keep the insoluble residue for further examination.

(a) Add a few drops of silver nitrate solution; a white curdy precipitate, soluble in ammonium hydrate and insoluble in nitric acid, indicates the presence of chlorides.

(b) Add a few drops of barium chloride solution; a white precipitate, insoluble in hydrochloric acid, indicates the presence of a sulphate.

(c) Test the presence of a nitrate by the ring test with ferrous sulphate and sulphuric acid as directed before (p. 74).

(d) Test the presence of sodium, calcium, magnesium and iron by their respective tests.

(e) The residue insoluble in distilled water should be partly treated with dilute hydrochloric acid in a test tube fitted with a cork and delivery tube, and if any effervescence is noticed, the evolved gas should be passed into lime water and tested for CO_2 .

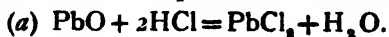
(f) Dissolve another portion of the residue in the least quantity of dilute hydrochloric acid and test the solution for calcium and magnesium by their respective tests.

CHAPTER XX

SIMPLE CHEMICAL PREPARATIONS

1. LEAD CHLORIDE

Take small quantities of litharge, PbO , and lead peroxide, PbO_2 , in separate test tubes add hydrochloric acid to each and boil. Observe that chlorine is evolved only in the latter case and lead chloride is formed in both, which when dissolved in boiling water and allowed to cool is deposited in fine crystals



2. LEAD NITRATE

Take some dilute nitric acid in a porcelain basin, add to it small quantities of lead monoxide or litharge, stir with a glass rod and warm the mixture. When the whole of the acid is neutralised and no more of litharge is dissolved, filter off the liquid and observe that the filtrate is neutral, *i.e.*, turns neither blue litmus red nor red litmus blue. Concentrate the solution by evaporation and allow to cool. Crystals of lead nitrate will appear.

3. COPPER SULPHATE

Use black copper oxide, and dilute sulphuric acid and apply the above method. The filtrate on evaporation will yield blue crystals of copper sulphate.

4. ZINC SULPHATE

Apply the same method using zinc oxide and dilute sulphuric acid.

5. SODIUM HYDRATE

Take some dilute solution of sodium carbonate and treat it with milk of lime. Warm the mixture and stir with a rod. After some time allow the precipitate of calcium carbonate to settle and test a few drops of the clear supernatant liquid with dilute hydrochloric acid; if no effervescence is noticed the whole of the sodium carbonate must have been changed to sodium hydrate. Filter and evaporate the dilute solution of sodium hydrate, thus formed, first in a porcelain basin and finally in a silver or nickel basin and heat to dull redness. A white residue of caustic soda will be found.

6. PURE SODIUM CHLORIDE

(i) Prepare a solution of sodium chloride by the neutralisation of dilute hydrochloric acid by a dilute solution of pure sodium hydrate, using pieces of red and blue litmus papers, spread on a porcelain tile, as indicators; the neutral product will neither redden blue litmus nor turn red litmus blue. Evaporate this neutral solution and allow to cool; crystals of sodium chloride will appear.

(ii) Prepare a saturated solution of common salt (brine) and filter. Collect the clear filtrate in a beaker

and pass hydrochloric acid gas into it (Fig. 68). The hydrochloric acid gas is conveniently generated by dropping strong hydrochloric acid from the tap funnel

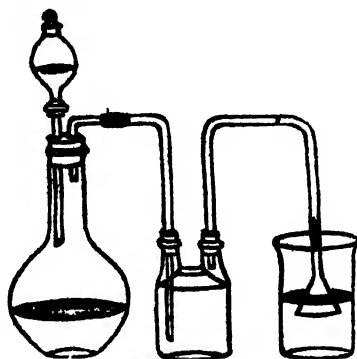


Fig. 68.

on strong sulphuric acid contained in the flask. As sodium chloride is insoluble in hydrochloric acid solution, it will appear as crystals after the passing of the gas for some time. Use an inverted funnel for passing the gas into the liquid as ordinary delivery tubes are easily blocked up by the substance precipitated. Allow the crystals to settle and separate them from the mother liquor by filtration. Wash with strong hydrochloric acid while on the filter and spread on a flat porcelain basin. Heat the basin gently over an asbestos board to drive off moisture and hydrochloric acid.

APPENDIX I

TABLE OF ATOMIC WEIGHTS

O = 16

O = 16

Aluminium	Al	27.10	Iron	Fe	55.85
Antimony	Sb	120.20	Lead	Pb	207.10
Arsenic	As	75.00	Magnesium	Mg	24.32
Barium	Ba	137.37	Manganese	Mn	54.93
Bismuth	Bi	208.00	Mercury	Hg	200.00
Boron	B	11.00	Nickel	Ni	58.68
Bromine	Br	79.92	Nitrogen	N	14.01
Cadmium	Cd	112.40	Oxygen	O	16.00
Calcium	Ca	40.09	Phosphorus	P	31.00
Carbon	C	12.00	Platinum	Pt	195.00
Chlorine	Cl	35.46	Potassium	K	39.10
Chromium	Cr	52.10	Silicon	Si	28.30
Cobalt	Co	58.97	Silver	Ag	107.88
Copper	Cu	63.57	Sodium	Na	23.00
Fluorine	F	19.00	Strontium	Sr	87.62
Gold	Au	197.20	Sulphur	S	32.07
Hydrogen	H	1.008	Tin	Sn	119.00
Iodine	I	126.92	Zinc	Zn	65.37

APPENDIX II

TENSION OF AQUEOUS VAPOUR

Degrees C	Tension in millimetres	Degrees C	Tension in millimetres
15	12.699	28°	28.101
16'	13.536	29°	29.782
17°	14.421	30'	31.548
18°	15.357	31'	33.406
19'	16.346	32'	35.359
20'	17.391	33°	37.410
21°	18.495	34°	39.565
22	19.659	35°	41.827
23°	20.888	36°	44.201
24°	22.184	37°	46.691
25°	23.550	38°	49.302
26	24.988	39°	52.039
27°	26.505	40°	54.906

APPENDIX III
SIRENGTH OF SULPHURIC ACID OF DIFFERENT
DENSITIES—(*Lünge*)

Sp. gr. at 15°C.	Grams H_2SO_4 per litre	Percentage of H_2SO_4 by weight.
1.200	328	27.32
1.300	510	39.19
1.400	702	50.11
1.500	896	59.70
1.550	996	64.26
1.600	1099	68.70
1.650	1204	72.96
1.700	1312	77.17
1.720	1357	78.92
1.740	1404	80.68
1.760	1451	82.44
1.780	1504	84.50
1.800	1565	86.92
1.805	1581	87.60
1.810	1598	88.30
1.815	1621	89.05
1.820	1639	90.05
1.825	1661	91.00
1.830	1685	92.10
1.835	1717	93.56
1.840	1759	95.90
1.840	1816	98.72
1.841	1784	96.30
1.841	1808	98.20
1.8385	1826	99.31

APPENDIX IV
STRENGTH HYDROCHLORIC ACID OF DIFFERENT
DENSITIES—(*Lunge*)

Sp. gr. at 15°C.	Grams HCl per litre	Percentage of HCl by weight
1.110	243	21.92
1.115	255	22.86
1.120	267	23.82
1.125	278	24.78
1.130	291	25.75
1.135	303	26.70
1.140	315	27.66
1.145	328	28.61
1.150	340	29.57
1.155	353	30.55
1.160	366	31.52
1.165	379	32.49
1.170	392	33.46
1.175	404	34.42
1.180	418	35.39

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